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Technical Paper 296

DEPARTMENT OF THE INTERIOR
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SIZE AND CHARACTER OF GRAINS OF NONMETALLIC MINERAL FILLERS

BY
W. M. WEIGEL

WASHINGTON
GOVERNMENT PRINTING OFFICE
1924

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First edition, March, 1924.

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SIZE AND CHARACTER OF GRAINS OF NONMETALLIC MINERAL FILLERS.

By W. M. Weigel.

INTRODUCTION.

Mineral fillers, as the term is used in this paper, are those nonmetallic minerals that, after being pulverized, are used in the preparation of various manufactured articles. Kaolin, whiting (applied broadly to chalk and to ground limestone and marble, silica, ocher, mica, talc, barite, slate flour, graphite, and diatomaceous earth are the chief mineral fillers. Different forms of some of these minerals are employed; as, for example, silica may be ground quartz, or pumice, or tripoli, the fine rounded grains resulting from the weathering of chert.

Each mineral has properties that make it suitable for certain uses. These properties are color, specific gravity, the nature of the surface, and the shape and, probably of greatest importance, the size of the particles. Most fillers are used essentially as inert substances to occupy space, fill voids, or impart color, but in some lines of manufacture they have an important but not easily understood effect on the physical properties of the finished article. For example, ground barite and marble, aside from their specific gravity, have particles that are much alike in appearance and shape, and yet the properties they impart to a rubber compound are quite different. Of chief importance in the comparison of fillers from the same mineral, and to a less degree of fillers from different minerals, are the average particle size, the variation in size from maximum to minimum, and the proportionate amounts of the different sizes; the shape of the grains ranks second and applies more especially to the comparison of fillers from different minerals.

Although fillers are used in a great number of industries, the bulk of the material goes into the following products: Paper, wallpaper, prepared roofings, rubber, paint, linoleum, oilcloth, foundry facing, plastic cements, oxychloride cement, artificial stone, polishes and scouring compounds, matches, dressing and dusting powders, various textile products, such as window shade cloth, and phonograph records.

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The data given in this paper are published by the Bureau of Mines as the result of an investigation made by its division of mineral technology.

ACKNOWLEDGMENTS.

Acknowledgments are due to the many companies who furnished samples of fillers; and especially to G. St. J. Perrott and S. P. Kinney, of the Bureau of Mines, for helpful suggestions and criticisms, as they had done somewhat similar work.[1] H. F. Anders, chemist at the Southern experiment station of the Bureau of Mines, made the chemical analyses given.

METHODS OF DETERMINING GRAIN SIZE.

SCREEN ANALYSIS.

Ordinary screen analysis is of little value except in determining the small proportion of coarse grains in some fillers; it gives no idea of the true average size of grains. The finest screens procurable have 300 and 350 meshes to the inch, with a nominal opening equal to 40 to 50 microns (1 micron equals 0.001 mm.), and all high-grade fillers, except a few special coarse ones, have an average grain size less than 15 microns. Moreover, satisfactory screens of the finer meshes are difficult to procure; even with careful selection of the wire cloth the size of the individual openings will vary as much as 30 to 40 per cent.

OTHER METHODS.

A number of different methods have been proposed, and have been used to some extent for special fillers. Some of these methods are based on empirical factors expressing the degree of fineness relatively without giving actual numerical values of the size of individual grains.

Different forms of air analysis are often used to separate the product into a number of fractions, the grains in the fractions being measured with a microscope fitted with a filar micrometer eyepiece. J. C. Pearson and W. H. Sligh [2] describe such an analyzer as designed by the Bureau of Standards for determining the fineness of Portland cement. Compared with most fillers, cement is coarse grained, and it is yet to be seen whether satisfactory air separations of materials finer than 10 microns can be made. For some fillers, notably kaolins in which more than half the grains may be less than 5 microns in

[1] Perrott, G. St. J., and Kinney, S. P., The meaning and microscopic measurement of average particle size: Jour. Am. Ceramic Soc., vol. 6, Feb., 1923, pp. 417–439.

[2] Pearson, J. C., and Sligh, W. H., An air analyzer for determining the fineness of cement: Bureau of Standards Technol. Paper 48, 1915, 74 pp.

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diameter, several fractions below this size would be necessary if reliable comparisons are to be made between similar materials.

As most fillers are insoluble in water, the products are often elutriated in a series of rising currents of water with different velocities. Elutriation gives fairly accurate results within its limits and separates the coarser particles fairly well, but the shape of the grains has an important effect, and in the slower currents of water it has been observed that the velocities are not equal over the entire area, as convection currents are set up through differences in temperature between the air and the surface of water and container. Moreover, some fillers do not yield well to deflocculating or dispersing reagents, and the finer sizes will be found in the coarser fractions. This is clearly shown in Plates III, *b*, and IV, *a* (p. 44), which show elutriated fractions of a kaolin. After the various elutriated fractions are obtained, their separate average sizes must be determined by calculation from an empirical formula or by actual count and measurement under the microscope. Calculation of the size of particles of each fraction is usually made by Schöne's[3] formula, which is as follows:

$$D=0.0518\frac{V^{0.636}}{S-1}, \text{ in which}$$

V=velocity of flow in millimeters per second.

D=maximum diameter in millimeters of spherical grain carried over by velocity V.

S=specific gravity of the substance.

To find the average size of the fraction it is necessary to ascertain the numerical average of the maximum inflowing and outflowing grains. This may be much in error, for there may be a much larger proportion of the grains near one or the other of the limiting sizes. This is especially true for the product in the first can and in the overflow, for the overflow may be made up mostly of particles nearer zero diameter than the maximum discharged by the last can. One of the main objections to water elutriation is the time required—for some substances 36 to 100 hours for a good separation.

The microscopic measurement and count of each fraction is described on the succeeding pages. A method proposed by Green[4] is especially applicable to very finely divided pigments, such as zinc oxide, and to particles whose maximum and minimum sizes do not cover a wide range. This method consists essentially in making a photomicrographic negative of the pigment, projecting the negative on a screen at a known magnification, and measuring the projection of the particles with a scale. With the proper facilities and procedure

[3] Schurecht, H. G., Elutriation tests on American kaolins: Jour. Am. Ceramic Soc., vol. 3, May, 1920, p. 359.

[4] Green, Henry, A photomicrographic method for the determining of particle size of paint and rubber pigments: Jour. Franklin Inst., vol. 192, Nov., 1921, p. 637.

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results should be as accurate as is possible at present for the class of materials to which the method is adapted; that is, materials whose maximum and minimum grains vary so slightly in size that one negative, or at the most two or three, will represent a fair average, and that all the grains can be brought sharply into focus with one objective. This can not be accomplished with all the common nonmetallic fillers. Plate XIII, *a* (see p. 44), shows how impossible would be the making of a photomicrograph that sharply defines all grains from the largest to those 1 micron and less in diameter.

METHODS USED.

In determining the grain size of a number of representative fillers two methods were used in order to check results and to find which method was the easier and the more accurate. By the first method the sample was elutriated into five fractions and the average size of each fraction determined from microscopic count and measurement. The average size of the total sample was then calculated from that of the fractions. By the second method a microscopic count and measurement of a representative sample of the product was made without any previous separation into groups of sizes. The procedure as to measurement and count was the same in both methods.

ELUTRIATION.

The apparatus, the general arrangement of which is shown in Plate I, consisted of two sets of four tinned-copper elutriating cans with conical bottoms of the Schulze form as modified by R. T. Stull.[5] The first can was 2.25 inches in diameter; the second, 3.156 inches; the third, 5.47 inches; and the fourth, 9.5 inches. The height of the sides of all the cans was 7 inches. The manipulation of the apparatus is fully described by Schurecht,[6] and his methods were in general followed. To keep material from settling along the sides of the cone, however, part of the water was siphoned off and the contents of the can agitated by blowing air through the thistle tubes twice daily instead of only once. The flow of water was adjusted so that after the first few hours the velocity in the cans was as follows: Can 1, 0.393 mm. per second; can 2, 0.200; can 3, 0.067; can 4, 0.022. For barite these amounts were increased by 50 per cent. In constructing a new set of cans it would be advisable to make the height of the third and fourth cans at least one and one-half or two times their diameter and thus insure a more uniform rising current over the area.

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[5] Supervising ceramist, U. S. Bureau of Mines.

[6] Schurecht, H. C., Elutriation tests on American kaolins: Jour. Am. Ceramic Soc., vol. 3, May, 1920, pp. 355–378.

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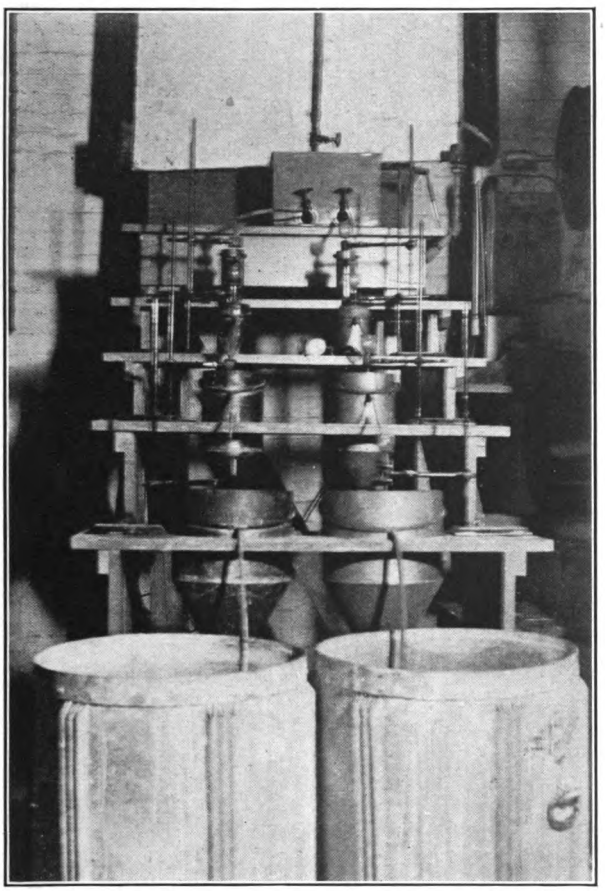

ELUTRIATION APPARATUS, SOUTHERN EXPERIMENT STATION, BUREAU OF MINES.

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Fifty grams of the material to be used was weighed out and screened wet through a set of Tyler standard testing sieves; the minus 200 mesh was then charged to the elutriator. Clay samples were first blunged, or agitated, with water and the necessary amount of alkali for deflocculation for one and one-half hours in a 2-quart Dazey churn. The coarse material that remained on the screens was dried and weighed to determine the respective amounts, and as these are small for most fillers the numerical average of the screen openings between which they fell was taken as the mean diameter.

The overflow from the fourth or last can was caught in much larger cans. (Pl. I.) The fine material in the overflow settles very slowly, sometimes taking several days. Therefore to obtain samples without delay small amounts were caught at regular intervals and combined to make up an average from which the microscopic slides were prepared without dewatering. The weight of the overflow was taken as the difference between the amount used and the combined weight of the first four fractions.

As the samples of fillers tested were elutriated with the idea of preparing groups of sizes within a fairly narrow range for microscopic count and measurement, the calculated sizes based on the velocity of rising current were not determined for the respective minerals, as the method of calculating average size was not based on the mean of the maximum and the minimum in each can. Table 1 shows the relation between the theoretical average of each fraction, calculated by Schöne's formula for grains with specific gravity of 2.72, and the average size, as determined by microscopic count and measurement and calculated on the basis of specific surface. (See "Calculation of average particle size," p. 20.)

TABLE 1.—*Comparison of average diameters*[a] *of elutriated fractions calculated by Schöne's formula, and microscopic count and measurement.*

Fraction	1			2			3			4			5		
Material.	Maximum.	Minimum.	Average.	Maximum.	Minimum.	Average.	Maximum.	Minimum.	Average.	Maximum.	Minimum.	Average.	Maximum.	Minimum.	Average.
Schöne's formula, sp. gr. 2.72	.....	.....	45.4	.....	.....	13.7	.....	.....	8.0	.....	.....	4.0	.....	.....	1.3
Clay, 10 samples	54.3	41.4	47.8	36.7	21.1	28.3	15.5	6.6	11.6	9.0	2.6	5.9	4.4	1.7	2.6
Whiting, 2 samples	38.1	25.1	31.6	20.2	12.5	16.3	11.6	7.2	9.4	6.2	4.2	5.2	3.7	2.6	3.2
Ocher, 2 samples	64.2	47.0	55.6	32.0	21.3	26.6	17.8	12.4	15.1	7.7	7.4	7.6	3.7	1.8	2.7
Slate flour, 2 samples	46.8	45.5	46.1	29.7	26.1	27.9	16.2	13.1	14.7	6.7	6.0	6.4	3.7	2.1	2.9
Silica, 1 sample	.....	.....	42.2	.....	.....	16.8	.....	.....	13.8	.....	.....	6.4	.....	.....	2.0
Talc, 2 samples	34.7	30.0	32.3	24.8	18.7	14.2	9.7	8.9	9.3	7.8	5.4	6.6	4.7	3.5	4.1

[a] Diameters are in microns.

The error of assuming that each fraction of minerals of the same specific gravity, or even of the same mineral, has a fixed average

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particle size can be shown from this table. The two samples of slate flour elutriated were prepared from the same slate. The first was the rather coarse product as received from the manufacturer; it had been ground in a tube mill. The second sample of flour was given an additional grinding in the laboratory in a pebble mill. The average size of the reground product was about one-half that of the original. The maximum size given in Table 1 for each of the fractions refers to the coarser product. The difference is especially evident in the overflow, or fifth, fraction. The maximum and minimum grains of the respective fractions from each product should be the same, but in the reground material the proportion of minimum grains is much greater. This also held good for clays; in general, the maximum values in the table belong to the coarser clays and the minimum values to the finer ones.

TOTAL SAMPLE.

For the microscopic measurement and count of the grains of a filler two methods are available: First, accurate measurement of the diameters of a comparatively few grains with a filar micrometer eyepiece; second, the less accurate estimation of a very large number of grains in a large number of fields. For measuring a material of uniform size in which the grains are comparatively large (5 microns or more) the first method probably has advantages. The range of size of all nonmetallic fillers is, however, wide, and most of them are so small that they can not be measured accurately with ease. The larger grains are few, but on account of their greater volume influence the average size in much greater proportion to their number than the finer grains. Any method must take into account of all sizes of grains. Since the largest grains are so few, it may be necessary to examine 50 to 100 fields before one is encountered. This condition will often be found when the field is as thick or crowded as possible and yet each grain is free from contact with others, a necessary condition for measurement.

The second method mentioned above was proposed and used in principle by Perrott and Kinney[7] for determining the average particle size of pulverized coals, and was modified to suit mineral fillers. Sound technique in the use of a microscope, including the preparation of slides, is necessary to success. But with these qualifications, and a little practice, checks close enough for this class of material, and probably as close as two separate samplings would be, can be obtained.

The method of total sample was employed as given in the following pages to determine the average grain size of all the fillers, and

[7] Perrott, G. St. J., and Kinney, S. P., The meaning and microscopic measurement of average particle size: Jour. Am. Ceramic Soc., vol. 6, Feb., 1923, pp. 417–439.

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elutriation and determination of the average size of the fractions was used on enough samples to show comparative results.

PREPARATION OF SAMPLES.

Twenty grams of well-mixed material is screened wet on a set of testing sieves to 200 mesh. If an appreciable amount remains on the sieves, it is dried and weighed and the percentage of the original determined. As these fractions are nearly always very small and influence the average size but little, the average size of each fraction is taken as the arithmetical mean of the size of screen openings between which it falls.

Samples of clays are blunged in a Dazey churn for one hour before screening with 1,000 cubic centimeters of distilled water and enough NH_4OH to deflocculate the particles; then they are screened on 200 mesh. The plus 200-mesh material is discarded. Practically all clays used as fillers are washed or ground to pass 200, and this standard is taken to represent the material which would be marketed. The washing from the 200-mesh screen is diluted to about 2 liters and transferred to a tall jar, 5 inches in diameter and 12 inches high, and thoroughly agitated by blowing through it a rapid current of air by means of a tube that extends to the bottom. During this agitation several samples, enough in all to make 50 cubic centimeters, are withdrawn with a pipette, placed in a 1-inch by 10-inch test tube, and reserved for microscopic count. It is believed that unless precautions are taken during this sampling to keep the suspension violently agitated and to take first a large sample of at least 20 grams, there is likely to be more chance for error in the sampling than in the final count.

Putting less than 1 milligram of dry powder on a slide and working it out to the proper density will not give a fair average of the material where the limiting sizes are so far apart.

PREPARATION OF SLIDES.

The sample in the test tube is diluted by adding distilled water, shaking, and pouring off the surplus until the desired turbidity, which may be described as cloudy or very slightly milky, is obtained. The proper degree of dilution can be determined only by experience. The tube should be not more than one-third full. A pipette is run to the bottom and the contents are agitated by blowing air through the tube; then a few drops are quickly drawn into the pipette and placed on the slide. The solution on the slide should cover an area about 20 mm. in diameter. The slide is carefully placed in an air bath and dried at 105° C. The film of grains on the slide will hold well and permit handling with ordinary care.

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The density should be such that all grains are separated from each other and may be counted without confusion. Drying on a hot plate is unsatisfactory as it gives an uneven film. Plate VIII, *a* (p. 44), shows a slide of convenient density for measurement and count, while Plate II, *b* (p. 44), is too crowded for satisfactory work. In general, it has been found that, except with clays, better dispersion of the particles is obtained by the use of distilled water alone than with any deflocculating agent. A drop or two of NH_4OH added to the test tube will increase the dispersion of the particles in most clay samples. Slides that show aggregates of fine grains or flocculated material are worthless for counting, and often several slides must be prepared before a satisfactory one is obtained.

For the preparation of slides from very fine-grained pigments, as ZnO, Green[8] works out the material on the slide with redistilled turpentine, using a glass rod. As the grains are so fine and the surfaces of the slide and rod not optically true, little or no crushing results. This method can not, however, be applied to the fillers considered in this paper, as crushing or grinding of the larger grains is inevitable and gives low results.

Slides used measured 50 by 75 mm. The ordinary slide, 25 mm. wide, is too small to provide a large enough area of film for the number of fields that must be counted.

The index of refraction of most of the nonmetallic minerals that are used as fillers falls between 1.50 and 1.60, or close to that of the ordinary mounting media, Canada balsam and gum damar. For this reason, the very fine particles, with the exception of colored pigments, such as the ochers, are difficult to see under the microscope when so mounted, as they are transparent and colorless. It was found much easier and better to use the slide uncovered for all fillers except those that had a large proportion of grains less than 1 micron in diameter, such as clays. Objectives corrected for uncovered objects should be used. Where grains less than 1 micron must be counted a 1.9 or 2 mm. oil-immersian objective is necessary and a cover glass must then be employed.

Glycerin was found to be a satisfactory mounting material for many of the nonmetallic minerals. Its index of refraction is 1.47 and it is viscous enough to hold the grains stationary. For clays and talcs, whose very fine grains are particularly difficult to count, methylene iodide, with an index of refraction of 1.74, is used. Since it disperses light rather strongly and creates diffraction rings around the particles, it is, however, used only when necessary.

[8] Green, Henry, A photomicrographic method for the determination of particle size of paint and rubber pigments: Jour. Franklin Inst., vol. 192, Nov., 1921, p. 640.

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A rectangular cover glass, 35 by 50 mm., is employed. A drop of glycerin or methylene iodide is placed on the center of the slide and the cover dropped in place and worked down with slight pressure and a sliding or rotary motion. The excess liquid is removed around the edges with filter or blotting paper, and with a camel's-hair brush a ring of melted paraffin is run around the cover, cementing it in place. This acts immediately, and while not as strong as Brunswick black or other cements, allows the slide to be used at once, answers all purposes, and has no effect on the methylene iodide. Care must be taken to use the minimum of mounting material, for if any of the grains are washed from under the cover the results can not be trusted. This is one advantage of using uncovered slides when possible.

In preparing slides of elutriated fractions less care is necessary to get a fair sample, as each fraction is sized to much closer limits. For this work about 0.5 gram is placed in a large test tube, thoroughly shaken with distilled water, and then handled the same as the 50-c. c. sample taken from the screen washings. This procedure applies to the finer fractions. For the first fraction, especially, a fair average in a water suspension was difficult to get, as the coarse grains settled too rapidly. Equally good results were obtained by placing about 0.5 milligram of the dry sample on the slide, adding a few drops of water, and carefully working the sample around with the end of a glass rod. With the larger grains no flocculation takes place. The fine particles eventually tend to coagulate in practically all liquid-mounting media, and it is best to make up the slide just before the microscopic examination, or at the most not over two to three days before.

COUNT AND MEASUREMENT.

A petrographic microscope should be used. Then, employing the crossed nicols, one can often distinguish easily between floccules and individual grains; without nicols the distinction at times may be difficult. The ocular No. 10 is fitted with a micrometer disk ruled in squares so that there are six to eight squares to a side of the total square inscribed within the circle of the field. One of these smaller squares is again subdivided. In the ocular used the large square was divided into seven each way and the central square was divided into five each way. In the photomicrographs reproduced, only the central part of the field is shown. Observing the field through this screen and knowing the value of the divisions, one can approximate closely the size of the individual grains. If the size of any particle is in doubt, the slide is moved to bring it under the finer divisions. In the actual count only the inner 25 large squares were used, as those in the outer row, being near the edges of the field, were distorted with high magnifications.

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The procedure followed is as follows: Beginning with the lowest power objective to be used and at one extreme edge of the slide, the field is looked over and the largest grain found and estimated, calling it the even size nearest which it falls; that is, if it averages 47 microns it is called 50 microns. Starting at the upper left corner of the 25 central squares the field is gone over square by square and all grains that fall between 45 and 55 microns are counted. The count is recorded as so many grains of 50 microns diameter. Beginning again, the grains between 45 and 35 microns are counted and recorded as 40 microns, and so on to the smallest size that can be determined accurately with the lowest power objective. The slide is then moved to a new field and the process repeated. In this way the whole slide is worked over, and as many fields counted as is considered necessary for obtaining a fair average.

A mechanical stage is convenient but not necessary as long as the field is covered in a fairly systematic manner. The method used was to work back and forth across the slide in both directions in approximately straight lines. Often the finer grains will be concentrated near the edges of the slide, and overcounting or undercounting these areas must be avoided. If there is any doubt a new slide must be prepared.

The objective is then changed to the next highest power and the group of sizes selected as falling best within its range are counted, and so on to the highest power objective and the finest grains. In counting 1-micron grains with the 4-mm. objective and 0.5-micron grains with the 1.9 or 2 mm. objective the task becomes laborious and the time needed will be greatly lengthened if the entire field is counted. If these sizes are well distributed in any one field only one-fifth of the area is counted, as follows: Beginning at the upper left corner the diagonal row of squares equal to one-fifth of the field is counted and the result multiplied by five in recording the count. In the next field the diagonal line is taken from right to left, and so on, alternating for each field.

As no two grains, even of the same material, are the same shape it is necessary to average the dimensions. That is, in the count each grain is mentally estimated as being equal to a cube whose edge or diameter equals the average of the two extreme dimensions of the observed particle. For example, if a grain is 38 microns long and 22 wide it is called a 30-micron grain. This is not strictly correct mathematically, but on account of the large number of grains of all shapes counted, the plus and minus errors compensate well enough for all purposes. It must be remembered that because of the irregular shapes of all grains, as seen in the photomicrographs of different fillers, no correct mathematical method of averaging can be made.

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The estimate made as above assumes that the thickness of the grains equals the average observed diameter. This is approximately true of most materials, and errors are probably compensated. For minerals of distinctly tabular or platy form, such as talc and mica, however, it becomes necessary to apply a correction, as the thickness of the grains that result from pulverizing is 50 per cent or less of the average observed diameters. Under these circumstances the procedure is to calculate the average diameters in the usual way and apply the correction to the final average. This gives the same result and is simpler than counting each grain as observed. The method of correction is shown under "Calculation of average particle size," p. 20.

MAGNIFICATION USED AND COMPARISON OF FIELDS.

For the coarser grains down to 30 microns, a 16-mm. objective was used, which with the No. 10 ocular gave a magnification of 100. With this, the values of the sides of the squares in the net micrometer were 30 microns for the smaller squares and 150 for the larger. An objective of higher power could be used, but its use is not advisable because the 16-mm. objective covers a larger area per field, and as the large grains are usually few it is desirable to cover the greatest area possible with the number of fields counted. For grains of intermediate size (30 to 10 microns) an 8-mm. objective was used that gave a magnification of 200. With this, the values of the micrometer were 15 microns for the small squares and 75 for the large. For the finer grains a 4-mm. objective was employed, giving a magnification of 430 and values of 7 and 35 microns, respectively, for the small and large squares of the net micrometer. With this combination grains were counted from 10 to 1 micron size. When it was necessary to estimate to less than 1 micron, a 1.9-mm. objective was used, giving a magnification of 950 and values for the net micrometer of 3.2 and 16 microns, respectively. The values of the divisions of the net micrometer were obtained by calibration with a stage micrometer with 0.01-mm. divisions.

The minimum size counted with one objective was again counted with the next higher objective and the results when reduced to equivalent areas were averaged. In this way calculations were checked and the larger area and number of grains counted in the one were balanced against the greater accuracy of the other. Results should agree closely.

There are few large grains; therefore, in order to get a fair average a large number of fields must be counted. For the intermediate sizes, with a much larger number of grains, fewer fields are needed to give an average, and for the finer grains fewer still, on account of their very large number and their more uniform distribution over

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the slide. In the counts made for this report, 80 fields were covered with the 16-mm. objective, 40 with the 8-mm., and 20 with the 4-mm. and the 1.9-mm. As a check, counts of one-half and double this number were made. In the count of one-half the number the results did not agree well, whereas in the other the results checked in all particulars as closely as any two samples of the same material. It is possible that in 80 fields the one largest grain might be missed, but the effect of not including it in the average would be negligible.

To combine the counts with the different objectives, they are reduced to grains per field for each objective and then multiplied by the ratio of the area of the fields to the area of the largest, or 16-mm. field. The 16-mm. objective field was 750 microns square; the 8-mm., 375 microns square; the 4-mm., 175 microns square; and the 1.9-mm., 80 microns square. The ratios of the three last to the first are as 87.89, 18.36, and 4, to 1.

To illustrate the entire procedure of tallying the count and measurement and reducing to one equivalent field Tables 2 and 3 are given, which show the count of ocher No. 1.

In the tables the arbitrary sizes selected are supposed to represent the grains of all sizes between the numerical averages of the arbitrary size and the limiting size next larger and next smaller. When elutriated fractions were being tabulated 20 fields of each were counted. This number is large enough, because the range of sizes is limited. Repeated trials showed that counting 10 fields yielded data that were not constant; whereas counting 40 fields showed little improvement over counting 20 in uniformity of results. The method of counting and tallying was the same as for the total count, except when the fewer fields were counted. One objective only is used for each fraction—the 16 mm. for the first, the 8 mm. for the second and third (unless this is contaminated with enough fines to make a count necessary, in which event the 4 mm. is used), the 4 mm. for the fourth, and the 4 mm. and the 1.9 mm. for the fifth, or overflow. Except for the last fraction, any reducing of the fields to equivalent areas is unnecessary, as each fraction is calculated alone.

CHECKING METHOD OF COUNT AND MEASUREMENT.

Accuracy, or ability to duplicate results of measurement, depends entirely on the method selected and the skill with which it is used. For any one method of calculation of the average size the accuracy is independent of the mathematical solution when the number of the respective sizes has been determined.

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TABLE 2.—*Screen analysis and microscopic count of ocher No. 1, part 1.*

Screen analysis.		
Mesh.	Per cent.	Average diameter.
+65	0.00	.....
−65+100	0.55	177
−100+150	1.18	125
−150+200	2.92	89
−200	95.35	[a] 4.8
	100.00	5.0

[a] As calculated from count.

−200 mesh with 16-mm. objective.												−200 mesh with 8-mm. objective.				
Field No.	Diameters in microns and number of grains.					Field No.	Diameters in microns and number of grains.					Field No.	Diameters in microns and number of grains.			
	80	60	50	40	30		80	60	50	40	30		30	20	15	10
1						41						1				1
2				1		42						2	1			4
3		1		1		43					1	3			2	3
4					2	44					2	4		1	4	7
5						45						5		1	6	8
6						46						6			4	4
7					1	47						7			1	4
8						48				1		8			1	1
9					1	49						9	1		2	6
10					1	50						10		3	4	5
11						51					1	11		1	3	7
12						52						12		1	2	5
13						53						13		1	2	4
14						54						14			3	5
15						55						15	1	1	1	4
16						56						16			2	2
17						57				1	2	17			2	2
18					3	58						18			1	1
19						59						19				
20				1		60						20				
21					1	61						21				
22						62					1	22				
23					3	63			1		1	23				2
24			1			64						24			2	2
25						65						25		1	2	3
26						66					1	26				3
27				1	1	67						27			1	3
28						68						28			2	4
29						69						29	1			2
30						70					1	30		2	3	4
31						71						31		1	2	1
32					1	72						32		1	4	8
33						73						33			1	1
34					1	74					1	34			2	2
35						75						35			1	1
36						76						36				3
37						77					1	37			2	3
38						78						38		1	1	4
39						79						39		1		
40						80				1		40				1
Total								1	2	7	27		4	16	63	120
Average per field								.012	.025	.087	0.337		0.1	0.4	1.57	3.0

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TABLE 3.—*Screen analysis and microscopic count of ocher No. 1, part 2.*

Field No.	−200 mesh with 4-mm. objective. Diameters in microns and number of grains.				−200 mesh with 1.9-mm. objective. Diameters in microns and number of grains.				
	10	7	5	3	5	3	1.5	1	0.5
1	1	.....	2	3	2	7	21	53	290
2	.....	3	5	15	1	9	19	32	290
3	2	3	6	17	1	4	10	35	490
4	.....	2	14	30	3	6	19	44	390
5	2	3	14	67	.....	3	18	44	360
6	.....	.....	.....	9	1	5	27	43	325
7	1	2	3	17	2	3	11	21	260
8	1	.....	.....	8	.....	3	11	31	260
9	1	3	3	25	.....	8	17	44	235
10	.....	.....	.....	7	1	7	11	36	185
11	1	1	4	17	.....	5	23	43	275
12	1	1	1	35	1	4	7	26	255
13	1	2	6	16	.....	1	10	29	220
14	3	4	5	32	1	2	15	27	240
15	1	1	4	27	3	9	12	38	325
16	1	4	7	28	1	4	17	48	190
17	2	6	5	34	1	6	18	24	290
18	2	4	14	64	1	4	10	22	195
19	.....	.....	4	38	2	6	15	44	205
20	.....	4	13	49	1	4	20	46	270
Total	20	43	110	538	22	100	311	730	5,520
Per field	1.	2.15	5.5	26.9	1.1	5	15.5	36.5	277.5

Multiplying number per field by ratio of area to 16-mm. field the result is as follows:

Field.	Diameter.												
	60	50	40	30	20	15	10	7	5	3	1.5	1	0.5
16-mm. field	0.012	0.025	0.087	0.337	.....	.....	.....	.....	.....	.....	.....	.....	.....
8-mm. field, per equivalent 16 mm.	.....	.....	.....	0.400	1.6	6.3	12.0	.....	.....	.....	.....	.....	.....
4-mm. field, per equivalent 16 mm.	.....	.....	.....	.....	.....	.....	18.4	39.4	101	494	.....	.....	.....
1.9-mm. field, per equivalent 16 mm.	.....	.....	.....	.....	.....	.....	.....	.....	97	439	1,362	3,208	24,389
Average number per 16-mm. field	0.012	0.025	0.087	0.368	1.6	6.3	15.2	39.4	99	466	1,362	3,208	24,389

Check counts by the same method that had been used in making the original count were made for the same slide, on different slides made up from the same sample, and with different samples of the same material. This procedure was followed both for total samples and for elutriated fractions. Either way good checks could be obtained, but sometimes the results from the same material by total sample against the average of the elutriated sample did not check well. It is believed that in the process of elutriation, when the

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weight of the last fraction is determined by difference, there is a chance for some loss in the handling and drying of the coarser fractions; as a result, the percentage of the last fraction appears larger than it really is and would make the average low. If the coarser fractions are contaminated with fines, there is a tendency to overlook the fine grains in counting with the lower power objectives and thus obtain a high resultant average for large grains. While these two possible sources of error may compensate, they are not always made in examining the same sample. A comparison of the results obtained by the total-sample method and the elutriation method is given in Table 4. Not all the samples determined by the total-sample method were elutriated.

TABLE 4.—*Average grain size of fillers, as determined by total sample and by elutriation methods.*

Name and No. of sample.	Average diameter by total sample.	Average diameter by elutriation.	Name and No. of sample.	Average diameter by total sample.	Average diameter by elutriation.
	Microns.	*Microns.*		*Microns.*	*Microns.*
Barite No. 1	9.8	8.5	Whiting No. 6	10.8	.....
Barite No. 5	7.3	7.5	Whiting No. 7	5.0	.....
Clay No. 4	2.9	2.6	Mica No. 4	13.8	.....
Clay No. 9	3.7	3.0	Mica No. 6	11.2	.....
Clay No. 10	5.4	4.2	Mica No. 9	8.7	.....
Clay No. 11	3.9	4.9	Ocher No. 1	5.0	5.9
Clay No. 12	5.4	4.9	Ocher No. 3	4.1	.....
Clay No. 13	5.8	6.4	Ocher No. 6	6.6	6.3
Clay No. 15	6.4	5.6	Slate flour No. 2	9.8	10.0
Clay No. 25	3.4	4.0	Slate flour No. 3	4.6	4.2
Clay No. 28	5.6	5.3	Silica No. 1	8.1	.....
Clay No. 32	3.3	3.6	Silica No. 6	5.2	.....
Clay No. 33	5.5	.....	Silica No. 8	5.9	6.2
Graphite No. 5	23.4	.....	Talc No. 2	12.4	14.5
Whiting No. 1	10.7	12.0	Talc No. 5	12.6	.....
Whiting No. 2	5.9	.....	Talc No. 7	8.7	.....
Whiting No. 4	5.7	4.8	Talc No. 8	11.8	11.5

RESULTS OF SOME CHECK COUNTS.

Clay No. 13.—Average diameter, as calculated from the count of the usual number of fields of the total sample, 5.60 microns; average diameter, as calculated from the count of twice the usual number of fields, 5.80 microns.

Clay No. 32.—Average diameter of the total sample, as calculated from the count of one slide, 3.40 microns; average diameter, as calculated from another slide made up from the same sample, 3.20 microns; average diameter, as calculated from the combined count of both slides, 3.30 microns.

Whiting No. 1.—First elutriated fraction, measured and counted with a 16-mm. objective; average diameter, 25.1 microns. Five months later another slide was prepared, measured, and counted with an 8-mm. objective; average diameter from second slide, 25.4 microns.

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Ocher No. 6.—Two slides were made up and the usual number of fields counted. Average diameter obtained for the first, 6.62 microns; for the second, 6.86 microns.

Slate flour No. 2.—Elutriated fraction 3; two slides prepared. First slide, one count of 20 fields; average diameter, 16.25 microns. Second slide, one count of 20 fields; average diameter, 16.25 microns. Same slide, another count of 20 fields; average diameter, 16.05 microns.

USE OF HIGH-POWER OBJECTIVE.

The necessity of using a high-power objective, 1.9 or 2 mm. oil immersion, was considered doubtful, and to check its effect on the average diameter, as determined by using only the 4-mm. objective, grains were counted to 1 micron with the 4-mm. objective, counting all grains of 2 microns and finer as 1; then the count was made with a 1.9-mm. objective, counting the 5, 3, 1.5, 1, and 0.5 grains, and the results as made by the two counts were calculated and compared.

In general it was found that when the percentage of very fine grains is large the highest-power objective should be used, as under these conditions the average diameter so determined may be greater or less than that obtained by counting to 1 micron only. If there are numerous fine grains, less than 1 micron in size, that may be missed at the lower magnification, the resulting average will be about the same or under the average obtained by the use of the 4-mm. objective alone. If all grains are visible and counted as 1 micron, the average obtained by subdividing this size into 1.5, 1, and 0.5 will exceed the average if they are grouped into the 1-micron size. With the coarser fillers, where the proportion of the fines is much lower, accurate enough results are obtainable with a 4-mm. objective.

It would seem, then, to be necessary to use the highest-power objective for estimating the finer-grained fillers averaging under 7 microns, especially the clays, ochers, and silicas that occur naturally in a finely divided state. For the coarser products, especially those prepared by mechanical grinding or reduction, the highest power is not needed for obtaining accurate results. Some of the results obtained by count and measurement of the finer sizes with the 4 and 1.9 mm. objectives are as follows:

Barite No. 5.—Counting to 1 micron with 4-mm. objective, average diameter is 7.35 microns; counting 1.5, 1, and 0.5 microns, with 1.9-mm. objective, average diameter is 7.27 microns.

Clay No. 4.—Counting to 1 micron, with 4-mm. objective, average diameter is 2.17 microns; counting 1.5, 1, and 0.5 microns, with 1.9-mm. objective, average diameter is 2.94 microns.

Clay No. 15.—Counting to 1 micron, with 4-mm. objective, average diameter is 6.44 microns; counting 1.5, 1, and 0.5 microns, with 1.9-mm. objective, average diameter is 6.40 microns.

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Whiting No. 1. Average of elutriated fractions.—Counting to 1 micron in fraction 5 with 4-mm. objective, average diameter is 11.99 microns. Counting fraction 5 with 1.9-mm. objective, average diameter is 11.86 microns.

Whiting No. 1. Total sample.—Counting to 1 micron with 4-mm. objective, average diameter is 10.7 microns; counting to 1.5, 1, and 0.5 with 1.9-mm. objective, average diameter is 10.10 microns.

Whiting No. 2. Total sample.—With 4-mm. objective, average diameter is 6.21 microns; with 1.9-mm. objective, average diameter is 5.83 microns.

Whiting No. 4. Total sample.—With 4-mm. objective, average diameter is 5.84 microns; with 1.9-mm. objective, average diameter is 5.66 microns.

Whiting No. 4. Fifth elutriated fraction.—With 4-mm. objective, average diameter is 2.63 microns; with 1.9-mm. objective, average diameter is 2.67 microns.

Whiting No. 6. Total sample.—With 4-mm. objective, average diameter is 11.26 microns; with 1.9-mm. objective, average diameter is 10.81 microns.

Whiting No. 7. Total sample.—With 4-mm. objective, average diameter is 5.11 microns; with 1.9-mm. objective, average diameter is 4.96 microns.

Ocher No. 1. Total sample.—With 4-mm. objective, average diameter is 4.60 microns; with 1.9-mm. objective, average diameter is 4.99 microns.

Ocher No. 2. Total sample.—With 4-mm. objective, average diameter is 4.30 microns; with 1.9-mm. objective, average is 4.11 microns.

Ocher No. 3. Average of elutriated fractions.—Counting to 1 micron in fifth fraction, with 4-mm. objective, average diameter is 5.70 microns; counting 1.5, 1, and 0.5 micron grains and using 1.9-mm. objective, average diameter is 6.29 microns.

Ocher No. 3. Total sample.—With 4-mm. objective, average diameter is 6.03 microns; with 1.9-mm. objective, average is 6.62 microns.

Slate flour No. 3. Total sample.—With 4-mm. objective, average diameter is 4.91 microns; with 1.9-mm. objective, average diameter is 4.64 microns.

Silica No. 1. Total sample.—With 4-mm. objective, average diameter is 8.09 microns; with 1.9-mm. objective, average diameter is 8.09 microns, an exact check.

Silica No. 6. Total sample.—With 4-mm. objective, average diameter is 4.80 microns; with 1.9-mm. objective, average diameter is 5.22 microns.

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Silica No. 8. Total sample.—With 4-mm. objective, average diameter is 5.50 microns; with 1.9-mm. objective, average diameter is 5.87 microns.

Talc No. 8. Total sample.—With 4-mm. objective, average diameter is 11.89 microns; with 1.9-mm. objective, average diameter is 11.80 microns.

These results show that of the nine samples of those materials, such as clay, ocher, and silica, in which the fineness of the individual grains is due to natural disintegration without mechanical reduction, one sample checks closely, two samples give lower average diameters when counted to 0.5 micron, while six give larger average diameters. Of the 10 fillers mechanically ground to a fine powder, one checks closely, one gives a larger average diameter when counted to 0.5 microns, while eight give smaller diameters. Also, the percentage of variation is less in this class of materials.

COMPARISON OF TOTAL SAMPLE AND ELUTRIATION METHODS.

If proper precautions are observed in making a slide that is truly representative of the unsorted material, it has been found that as good or better checks can be obtained than by elutriating into several fractions of graded sizes. The time required for a total sample determination, including the preparation of the sample and slide and the calculation, if a calculating machine is available, is from five to six hours. If a sample is to be graded by elutriation, an average of three days is first required for obtaining the fractions; then five slides must be prepared and nearly as many fields counted as in the total sample method. The time required for calculation will be a little longer, so that the total time may be estimated as four days.

After the average size is calculated by the total sample method it is easy to determine the percentage of the different sizes by weight or by surface of the different sizes, as is shown in Tables 10 and 11 (see pp. 32 and 34). This can also be done after the separate fraction have been calculated, but requires considerably more time as the percentage of each size of each fraction must first be determined and reduced to a percentage of the total sample. Probably less skill and training are required to obtain approximate results from elutriated fractions as results from the total sample count are more liable to be in error unless considerable care is exercised. The beginner will usually obtain higher results from the total sample.

AVERAGE PARTICLE SIZE.

MEANING OF TERM.

The average particle, or grain, size of a filler should be expressed as a numerical value that has a definite meaning in relation to some

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physical property. The numerical or arithmetical mean, obtained by dividing the summation of the product of the number of each size and its diameter by the total number, has no physical significance. The average particle size should, then, be based on the surface or volume, depending on which of these is more important in the particular line of work for which the filler is used.

The difficulty of defining or comprehending a rational value for average practicle size is made more evident by studying the photomicrographs of the total samples of fillers and by comparing the relatively large volume and few numbers of the large grains with the great number of the finer ones. This is exemplified by Table 5, which gives the relative numbers of the different-sized grains and was calculated from the average count per field of all clays. To simplify the comparison the largest grain considered was taken as unity.

TABLE 5.—*Average distribution of number of grains of different sizes in clays, with one grain of largest size as basis.*

Diameter.	Number of grains.	Diameter.	Number of grains.
Microns.		*Microns.*	
80	1.0	10	1,294
60	2.2	7	3,718
50	4.6	5	10,332
40	10.5	3	90,591
30	49.2	1.5	121,721
20	257	1	278,239
15	572	.5	2,738,726

In Table 5 the grains are assumed to be cubes and the volume of the one largest grain 512,000 cubic microns, while the volume of the total number of grains of the smallest size is only 342,340 cubic microns, but for filler purposes the effect of this volume in the form of 0.5-micron grains is much greater than that of the one large grain.

In the work for which most fillers are used the individual grains must first be so fine that they are invisible; second, so fine that they will not constitute a point of weakness in the material and thus start a rupture of it; and, third, so fine that maximum dispersion or diffusion will take place throughout the material. A 1-mm. cube would have little value as a filler. The same cube would make 1,000,000,000 cubes of 1 micron diameter, which evidently would disperse much more widely through the material and thus present an enormously greater surface for adhesion. It can be shown that for a unit weight of a given filler the adhesion, or bond, between the surface of the grains and the matrix varies inversely as the diameter of the grains. When the weight, or volume, of the larger grains is

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more important than the surface exposed an average diameter based on volume is proper.

As has been pointed out by Green,[9] the behavior, or effect, of fillers depends largely on their specific surface, by which is meant the surface exposed per gram of material, and this is inversely proportional to the average diameter of the grains.

For these reasons the average grain size as calculated for the fillers included in this investigation was based on specific surface. The average diameter, however, does not tell all that should be known about the grain size. The particles could all be of one size or have a wide limiting range without the fact being disclosed by a mere statement of the average, no matter how calculated, and yet the fillers could be quite different. In the tabulated count and measurement a means is available by which the proportions of the various sizes can readily be determined either by the per cent by weight of the respective sizes or by the total surface exposed. These factors have been calculated for the fillers considered and are given in Tables 10 and 11 (see pp. 32 and 34).

CALCULATION OF AVERAGE PARTICLE SIZE.

If one particle only be considered with its three dimensions respectively equal to l, b, and t, its average diameter may be calculated as follows:

$$(a)\ \frac{l+b}{2},\quad (b)\ \frac{l+b+t}{3},\quad (c)\ \sqrt{l\times b},$$

$$(d)\ \sqrt{\frac{2\,lb+2\,bt+\,2lt}{6}},\quad (e)\ \sqrt[3]{l\times b\times t}$$

(a) and (b) are arithmetical or statistical averages, (c) relates to the side exposed to view, (d) to the total surface, and (e) to the volume. In estimating thousands of grains of all shapes and forms strict adherence to any mathematical mean diameter of each grain is impossible and only an approximate average is taken; but in the calculation of the average size of the total number of grains the value depends on the weight given the factors of numbers, surface, and volume.

Perrott and Kinney[10] have fully discussed the derivation of the various formulas taking these factors into account, but part of this is repeated here.

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[9] Green, Henry, A photomicrographic method for the determination of particle size of paint and rubber pigments: Jour. Franklin Inst., vol. 192, Nov., 1921, p. 638.

[10] Perrott, G. St. J., and Kinney, S. P., The meaning and microscopic measurement of average particle size: Jour. Am. Ceramic Soc., vol. 6, Feb. 1923, pp. 417–439.

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The various formulas may be stated as follows:

1. Arithmetical or statistical mean, $d_{ave.} = \frac{\Sigma nd}{\Sigma n}$
2. Length mean, $d_{ave.} = \frac{\Sigma nd^2}{\Sigma nd}$
3. Average volume, $d_{ave.} = \sqrt[3]{\frac{\Sigma nd^3}{\Sigma n}}$
4. Surface mean, $d_{ave.} = \frac{\Sigma nd^3}{\Sigma nd^2}$
5. Weight mean, $d_{ave.} = \frac{\Sigma nd^4}{\Sigma nd^3}$

The derivation of formula 1 is evident, being simply the sum of each size multiplied by the number of grains of that size and divided by the total number. It has no physical meaning, and when the number of fine grains is great the coarser grains have little effect on the average diameter.

Formula 2, being the summation of the areas divided by the summation of the diameters, obviously gives a mean based on the surface observed, and the volume or total surface of the particle does not enter into the calculation.

Formula 3 is based on the average volume and may be stated as that diameter whose corresponding volume divided into the total volume equals the total number of grains.

If $d_{ave.}$ = average diameter
V = total volume
d_1, d_2, d_3, etc. = diameters of the different sizes
n_1, n_2, n_3, etc. = respective number of grains
then $V = \Sigma nd^3$
$d_{ave.}^3$ = volume of average grain, and
Σn = total number of grains

$$\Sigma n = \frac{\Sigma nd^3}{d_{ave.}^3}$$

$$d_{ave.} = \sqrt[3]{\frac{\Sigma nd^3}{\Sigma n}}$$

While giving results larger than the statistical and length mean, the few larger grains still do not exert much influence on the average diameter, which can not be considered as properly representing the average in a physical sense.

Formula 4 is based on the entire surface of the particle or on the total surface of a unit volume or weight of the material. It can be

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shown that if the particles are assumed as spheres or rectangular parallelopipeds

$$\text{that } d = \frac{6}{S\rho} \qquad (a)$$

in which d = diameter of particle in microns.[11]

S = "specific surface" or total surface in square meters per gram of material.

ρ = specific gravity.

The general equation for average particle size may be given as $d_{ave.} = \Sigma y d$, in which

y = per cent of particles of diameter d calculated as percentage of the total by number, length, surface, or volume.

If y = weight per cent of particles d and specific surface s then

$$y = \frac{nd^3}{\Sigma nd^3}, \text{ and}$$

$$S = \Sigma(ys) = \frac{\Sigma(nd^3 s)}{\Sigma nd^3}$$

If $d_{ave.}$ is average diameter, equation (a) may then be written

$$d_{ave.} = \frac{6}{S\rho} = \frac{6}{\Sigma(ys)\rho} = \frac{6}{\rho}\left(\frac{1}{\frac{\Sigma(nd^3 s)}{\Sigma nd^3}}\right)$$

where n = number of particles of diameter d.

Substituting for s, its value $\frac{6}{d\rho}$,

$$d_{ave.} = \frac{6}{\rho}\left(\frac{1}{\frac{\Sigma nd^3 \times \frac{6}{d\rho}}{\Sigma nd^3}}\right) = \frac{\Sigma nd^3}{\Sigma nd^2}$$

As the average diameter is a linear dimension, independent of weight, and as the specific gravity has been cancelled out, the average diameter calculated by the above formula may be considered as based on specific surface per unit volume.

The results obtained by this method seem to represent more nearly the value of finely divided, nonmetallic minerals for filler use, and the average diameters given were all calculated by it.

To apply this method to the calculation of the average diameter of a number of various sized grains, the values for the different observed diameters may be arranged in a table of the following form:

[11] If the particle is a rectangular parallelopiped, d is assumed to be the harmonic mean of the three dimensions, or $d = \frac{3\,l\,b\,t}{lb + lt + bt}$

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Values for different observed diameters.

Column 1.	Column 2.	Column 3.	Column 4.	Column 5.	Column 6.	Column 7.
d_1=observed diameter in microns.	n=number of grains counted.	d_1^3	nd_1^3=relative volume or weight of grain of size d_1.	$\frac{\Sigma nd_1^3}{\Sigma nd^3}$=each item of column 4 divided by sum of column 4.	S=specific surface for size $d_1=\frac{6}{d_1\rho}$	$S\times$per cent=product of column 5 by column 6.

The sum of column 7 equals total specific surface, but

$$d_{ave.}=\frac{6}{S\rho}=\frac{6}{(\Sigma \text{ column } 7)\rho}$$

It can be shown that the same result is obtained by dividing each item of column 5 by its corresponding diameter in column 1, or, expressed as an equation:

$$\Sigma\frac{\frac{nd^3}{\Sigma nd^3}}{d}=\frac{1}{d_{ave.}}, \text{ the reciprocal of the average diameter.}$$

This method is convenient for calculating the average size when the percentages of groups of average-size grains are given instead of numbers of grains, as in elutriated fractions, and in combining the average of microscopic count and measurement of minus 200 mesh with the coarser screen sizes. Results are arranged for calculation as follows:

Fraction number.	Per cent by weight of fraction.	Average diameter of fraction.	$\frac{\text{Per cent.}}{\text{Average diameter of fraction.}}$

$$d_{ave.}=\frac{1}{\Sigma\frac{\text{per cent}}{\text{ave. dia. of fraction}}}=\text{reciprocal of sum of last column.}$$

When the number of grains of each size is known, the most convenient method of calculation is that illustrated in the following pages, where a sample calculation is given.

Formula 5 is based on the weight or volume of the particles and gives average diameters that are larger than those calculated by the other methods.

In the general equation for average particle size

$$d_{ave.}=\Sigma yd$$

y_1 = per cent of particles of diameter d_1 figured as percentage of the total by volume

$$=\frac{n_1 d_1}{\Sigma nd}$$

substituting value of y,

$$d_{ave.}=\Sigma\left(\frac{nd_3}{\Sigma nd^3}\times d\right)=\frac{\Sigma nd^4}{\Sigma nd^3}$$

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In the calculation the fourth power of each diameter is multiplied by its corresponding number of grains, and the sum is divided by the sum of the cube of each diameter multiplied by its number of grains. To obtain the average of a number of groups as elutriated fractions which are expressed as a percentage, the per cent by weight of each entire group is multiplied by the average diameter of that group in microns, and the sum of these products equals the average diameter. This is shown as follows:

The relative weight or volume of each size is nd^3 and $\frac{nd^3}{\Sigma nd^3}$ is per cent of that size. Multiply by Σd, and this becomes $\frac{\Sigma nd^4}{\Sigma nd^3}$, which is the formula for the average diameter, or if the per cent of each size be multiplied by its diameter and the sum taken it equals the average diameter.

To show the variation in results obtained by the use of the several formulas the count and measurement of the minus 200-mesh product of ocher No. 1, given in Tables 2 and 3, were calculated by the different methods, with the following results:

1. $d_{ave.} =$ 0.67 micron.
2. $d_{ave.} =$ 1.26 microns.
3. $d_{ave.} =$ 1.59 microns.
4. $d_{ave.} =$ 4.77 microns.
5. $d_{ave.} =$ 14.72 microns.

With a higher degree of uniformity in the size of the grains, the difference between the results obtained by the different formulas becomes less, until, if the grains are all of one size, the results are equal.

DETAILS OF CALCULATION OF AVERAGE DIAMETER.

To illustrate the method of procedure, the count and measurement of ocher No. 1, recorded in Tables 2 and 3, are carried through the calculation as follows:

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TABLE 6.—*Calculation of average diameter of ocher No. 1, —200-mesh product.*

Diameter, d.	Number of grains, n.	d^2.	nd^2.	d^3.	nd^3.
60	0.012	3600	43	216000	2592
50	.025	2500	62	125000	3125
40	.087	1600	139	64000	5568
30	.368	900	331	27000	9936
20	1.60	400	640	8000	12800
15	6.30	225	1417	3375	21262
10	15.20	100	1520	1000	15200
7	39.40	49	1931	343	13514
5	99.00	25	2475	125	12375
3	466.00	9	4194	27	12582
1.5	1362	2.25	3064	3.37	4590
1.0	3208	1.0	3208	1.00	3208
0.5	24389	0.25	6097	0.125	3049
			25121		119801

$$d_{ave.}=\frac{\Sigma nd^3}{\Sigma nd^2}=\frac{119801}{25121}=4.77 \text{ microns.}$$

Combining with +200-mesh fractions.

Fraction.	Per cent.	Diameter.	$1000\frac{\text{per cent.}}{d}$
+100	0.55	177	0.0311
+150	1.18	125	0.944
+200	2.92	89	0.3280
−200	95.35	4.77	199.8951
	100.00		200.3386

$$d_{ave.}=\frac{1000}{200.3386}=4.99 \text{ microns.}$$

The number of grains of each size per 16-mm. field is taken from the last line of Table 3 and used in Table 6, as indicated. The factor 1000 is introduced, as percentages are expressed in parts per 100, to eliminate unnecessary figures at the right of the decimal point.

Calculation of the average of the elutriated fractions is carried out in a similar manner. The average of each fraction is obtained in the same way as in a total count, as shown in the first part of Table 6; then the average of the fractions is obtained in the same way as in the second part of Table 6. The following condensed calculation illustrates the method. Take slate flour No. 2. A residue of 17.17 per cent remained on 200 mesh after all passed 65 mesh. The average of screen-sized material was then the mean of 65 mesh or 208 microns and 200 mesh or 74 microns, or 141 microns. Division of this residue into closer limits with intermediate screen sizes would be more exact, but have little effect on the average. Fraction from can 1, 28.95 per cent, average diameter, 46.75 microns; can 2, 1.58 per cent, average diameter, 29.67 microns; can 3, 14.74 per cent, average diameter, 16.25 microns; can 4, 15.18 per cent, average

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diameter, 6.69 microns; overflow, 22.38 per cent, average diameter, 3.72 microns. The tabulation of these results is as follows:

TABLE 7.—*Calculation of average of elutriated fractions.*

Fraction.	Per cent.	Average diameter.	$1000\times\frac{\text{per cent}}{d}$.
Plus 200	17.17	141.00	1.217
Can 1	28.95	46.76	6.191
Can 2	1.58	29.67	.532
Can 3	14.74	16.25	9.071
Can 4	15.18	6.69	22.690
Overflow	22.38	3.72	60.161
	100.00	.....	99.862

$$d_{ave.}=\frac{1000}{99.862}=10.01 \text{ microns.}$$

If the grains are decidedly flat or tabular, as are those of talc, mica, and graphite, the results as calculated from observed diameters will be high when the calculation is based on surface. The average diameter should then be such that its specific surface truly represents the specific surface of the material. It is possible to apply a correction to accomplish this. Assuming cubes, the surface of a cube is $6d^2$. If the cube is split one way, the resulting surface becomes $6d^2+2d^2$, and if split three times the surface becomes $6d^2+6d^2$ or $12d^2$. That is, if the cube be cut by a number of planes parallel to the observed side, the increased surface is equal to twice the number of resulting parallelopipeds, minus one, multiplied by the square of the diameter. If the average thickness of the grains is expressed as a percentage of the observed diameter, the cube so subdivided would then have a new surface, stated as follows:

$$\text{Surface}=6d^2+2\left(\frac{100}{\text{Per cent}}-1\right)d,^2$$

$$\text{if } \frac{100}{\text{per cent}} \text{ be represented by } c$$

$$\text{Surface}=d^2(4+2c)$$

and the ratio of the new surface to the old is as $(4+2c)$ to 6.

The new specific surface, S, then becomes:

$$S=\frac{4+2c}{6}\times\frac{6}{d_{observed}\times\rho} \text{ and}$$

$$d_{corrected}=\frac{6}{S\rho}, \text{ and substituting value for } S$$

$$d_{corrected}=\frac{6}{6(4+2c)\rho}=\frac{6}{4+2c}$$

$$\text{or } d_{corrected}=\frac{6}{4+2c}\times \text{observed diameter.}$$

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In practice the average diameter is calculated in the usual way. Then the thickness of enough grains to obtain a fair average from largest to least is measured with the micrometer scale on the slow-motion focusing screw of the microscope, using a 4-mm. objective. The thickness of each size is calculated as percentage for that size, and the resulting percentages are averaged and used in the above formula. If the percentage by weight or surface of the different sizes is desired, the correction may be applied to each group of sizes and the average calculated from these. The result is the same. The given average diameters of graphite, talc, and mica were calculated by this method. The grains of graphite No. 5 had an average thickness of 50 per cent of the observed diameter; mica No. 4, 14 per cent; mica No. 6, 12 per cent; mica No. 9, 9 per cent; talc No. 2, 33.3 per cent; talc No. 5, 30 per cent; talc No. 7, 43.7 per cent; talc No. 8, 22 per cent.

PARTICLE SIZE OF FILLERS EXAMINED.

DESCRIPTION OF SAMPLES.

There were selected for examination a number of fillers that would show in some measure the difference in size of the grains of the same mineral and also the characteristics of different minerals. Most of the samples were from the Southern States and were therefore readily available for collection. The location from which the samples came and a brief description of their physical preparation and chemical analysis of the samples are given below. Clay samples were always prepared from crude material by crushing to 40 mesh, blunging, and screening through 200 mesh. The residue on 200 mesh was discarded.

Barite No. 1 is from Cartersville, Ga., is known as a regular off-color grade, and was prepared by dry grinding and air separation in a Raymond roller mill.

Barite No. 5 is a water-floated barite, not bleached; details of grinding and source were not obtained. The sample was supplied by a rubber manufacturer as the grade used by his concern.

Clay No. 4 is from near Iuka, Miss.

Clay No. 9 is from McIntyre, Ga., and is used entirely for filler purposes, mostly in the rubber industry.

Clay No. 10 is from McIntyre, Ga.

Clay No. 11 is a bauxitic clay from Toomsboro, Ga.

Clay No. 12 is in reality a bauxite, as shown by analysis, and was included to see how the fines produced by coarse grinding and slaking in water compared with clays. About 52 per cent remained as residue on 200 mesh.

Clay No. 13 is from Gordon, Ga.

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Clay No. 15 is from Langley, S. C.

Clay No. 25 is from Langley, S. C., but not from the same deposit as No. 15. The sample as received had been pulverized.

Clay No. 28 is an English china clay used largely in the manufacture of paper. It was evidently a washed product. The sample was supplied by the importer.

Clay No. 32 is from Cuthbert, Ga.

Clay No. 33 is from Butler, Ga. The sample received was not crude clay, but had been prepared by washing out the coarser grains.

Graphite No. 5 is the so-called dust screened from the refined flake graphite in the Alabama graphite district.

Whiting No. 1 is from Tate, Ga., and was prepared by grinding a white marble in a Raymond roller mill with an air separator.

Whiting No. 2 is from Cartersville, Ga., is known as commercial grade, and is prepared by dry grinding and air separation in a Raymond roller mill. Its original source is an oölitic limestone from Tennessee.

Whiting No. 4 is from Cartersville, Ga., and is prepared by the further wet grinding of whiting No. 2 in a Hardinge mill, followed by water classification, drying, and pulverizing. It is known as gilders' grade.

Whiting No. 6 is ground from white marble from Gantt's Quarry, Ala. The method of preparation not given.

Whiting No. 7 is English cliffstone whiting, supplied by an importer. The method of preparation is unknown, except that it is said to be bolted.

Mica No. 4 is from Canton, Ga., and was prepared by crushing and pulverizing a chlorite mica schist in a beater mill and screening through 200 mesh.

Mica No. 6 is from Biltmore, N. C., and was prepared by grinding scrap mica, dry, in Raymond pulverizers, and screening through 100 mesh.

Mica No. 9 is water-ground mica from Spruce Pine, N. C.

Ocher No. 1 is from Cartersville, Ga., and was prepared by log washing, sedimentation, drying, and pulverizing.

Ocher No. 3 is from near Iuka, Miss., from a property only partly developed. The sample is a so-called red ocher, being a dull brick-red, and is essentially a ferruginous clay rather than a true ocher.

Ocher No. 6 is a French ocher, and the sample was purchased in the market. The method of preparation is not known.

Slate flour No. 2 is from Fairmount, Ga., and was prepared by dry-grinding in a tube mill the screenings from the manufacture of roofing granules.

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Slate flour No. 3 is the same as No. 2, except that the laboratory sample was prepared by dry-grinding No. 2 in a laboratory pebble mill.

Silica No. 1 is from near Tredegar, Ala. It was the result of the weathering of chert. The material had been prepared by coarse crushing followed by screening to remove coarse lumps, and water classification.

Silica No. 6 is from Cleveland, Tenn. Several grades are prepared, but the sample counted was made up by coarsely crushing soft lumps of the crude material. These were screened on 40 mesh and the fines blunged with water, as in the preparation of clay, and screened on 100 mesh. Minus 100-mesh material was used for determining grain size.

Silica No. 8 is crude silica from near Iuka, Miss., and represents the weathering of a bed of chert. It was treated like silica No. 6.

Talc No. 2 is classed as extra-white grade. The crude talc came from near Marble, N. C., and was ground at Biltmore, N. C. Grinding was done dry in a disk crusher and a Fuller-Lehigh mill.

Talc No. 5 is from Chatsworth, Ga. It was broken to three-quarters inch in a jaw crusher and ground in a Fuller-Lehigh mill. This particular grade is known as B.

Talc No. 7 is from Winterboro, Ala. The crude talc was crushed and ground in a laboratory pebble mill. It is of the steatite variety, and the grains are much less tabular than those of talcs from Georgia and North Carolina.

Talc No. 8 is from Glendon, N. C. Strictly it is not talc but pyrophyllite. It has, however, all the physical characteristics of talc and is classed with talc so far as its use for fillers is concerned. The sample as received is known as grade A, and had been ground in a tube mill.

CHEMICAL ANALYSES.

The analyses given in Tables 8 and 9 were of samples as received or as marketed, with the exception of the clays and silicas 6 and 8. The clay samples were crude clay and the two silicas were washed products. Clays No. 13 and 33 represented washed material.

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TABLE 8.—*Chemical analyses of fillers.*

Sample.	$BaSO_4$.	$BaCO_3$.	SiO_2.	Fe_2O_3.	Al_2O_3.	CaO.	MgO.	Ash.	Volatile.	Fixed carbon.	Ignition loss.	Total.
Barite No. 1	96.15	0.69	1.35	0.92	0.24	0.0	0.0	.....	.....	.....	0.75	100.10
Barite No. 5	96.70	0.93	0.67	0.70	0.25	0.0	0.0	.....	.....	.....	0.80	100.05
Graphite No. 5	.....	.....	.....	.....	.....	.....	.....	46.59	7.30	46.11	.....	100.00
Graphite ash	.....	.....	24.77	3.20	17.98	0.20	.....	.....	.....	.....	.....	[a] 46.15

[a] Ash percentages calculated on basis of original graphite.

TABLE 9.—*Chemical analyses of fillers.*

Sample.	SiO_2.	Al_2O_3.	Fe_2O_3.	CaO.	MgO.	TiO_2.	MnO_2.	FeS_2.	K_2O.	Na_2O.	K_2O+Na_2O.	Ignition loss.	Total.
Clay No. 4	60.75	29.30	0.32	(a)	(a)	0.45	.....	.....	.....	.....	0.55	9.34	100.71
Clay No. 9	44.48	39.32	1.40	0.00	Trace.	1.08	.....	.....	.....	.....	0.40	13.35	100.03
Clay No. 10	48.67	36.75	1.10	0.00	Trace.	1.25	.....	.....	.....	.....	0.35	11.78	99.90
Clay No. 11	41.60	42.38	0.76	0.00	Trace.	1.14	.....	.....	.....	.....	0.48	14.12	100.48
Clay No. 12	12.76	58.57	1.58	0.00	Trace.	2.01	.....	.....	.....	.....	0.52	24.88	100.32
Clay No. 13	45.18	39.08	0.71	0.00	0.00	0.47	.....	.....	.....	.....	0.51	13.70	99.65
Clay No. 15	43.12	40.78	0.81	0.00	Trace.	1.05	.....	.....	.....	.....	0.58	13.79	100.13
Clay No. 25	41.06	42.60	0.95	0.00	Trace.	1.11	.....	.....	.....	.....	0.82	14.00	100.54
Clay No. 28	45.88	38.82	0.70	0.00	0.10	0.07	.....	.....	.....	.....	0.94	12.53	99.04
Clay No. 32	41.50	41.46	0.74	0.92	Trace.	1.50	.....	.....	.....	.....	0.46	14.15	100.73
Clay No. 33	44.22	39.32	0.77	0.55	Trace.	1.35	.....	.....	.....	.....	0.42	13.88	100.51
Whiting No. 1	1.52	[b] 2.43	.....	48.18	0.79	.....	.....	.....	.....	.....	.....	.....	.....
Whiting No. 2	0.78	[b] 0.89	.....	50.36	Trace.	.....	.....	.....	.....	.....	.....	.....	.....
Whiting No. 4	0.65	[b] 1.28	.....	50.31	Trace.	.....	.....	.....	.....	.....	.....	.....	.....
Whiting No. 6	0.58	0.90	0.06	51.13	0.05	.....	.....	.....	.....	.....	.....	.....	.....
Whiting No. 7	1.43	1.05	0.13	50.60	0.09	.....	.....	.....	.....	.....	.....	.....	.....
Mica No. 4	29.37	28.44	2.93	Trace.	27.97	.....	.....	.....	0.67	0.50	.....	10.50	100.38
Mica No. 6	45.89	36.79	1.57	0.00	1.43	.....	.....	.....	8.50	1.50	.....	5.04	100.72
Mica No. 9	44.26	38.83	1.07	0.00	1.65	.....	.....	.....	7.57	1.44	.....	4.60	99.42
Ocher No. 1	38.04	12.75	39.70	.....	.....	.....	1.01	.....	.....	.....	.....	6.40	97.90
Ocher No. 3	38.00	37.20	12.90	.....	.....	.....	Trace.	.....	.....	.....	.....	13.16	101.26
Ocher No. 6	40.65	21.60	26.16	0.62	Trace.	.....	0.10	.....	.....	.....	.....	9.73	98.86
Slate flour No. 2	54.19	26.29	6.55	Trace.	2.35	0.53	.....	0.58	2.85	2.22	.....	4.54	100.10
Silica No. 1	96.37	1.84	1.17	0.00	0.00	.....	.....	.....	.....	.....	.....	0.50	99.88
Silica No. 6	87.84	8.84	0.66	0.00	Trace.	.....	.....	.....	.....	.....	.....	2.43	99.77
Silica No. 8	95.44	2.23	1.14	0.00	0.00	.....	.....	.....	.....	.....	.....	0.51	99.32
Talc No. 2	58.70	5.67	0.64	0.00	31.92	.....	.....	.....	.....	.....	.....	3.30	100.23
Talc No. 5	55.18	3.16	6.06	(a)	29.02	.....	.....	.....	.....	.....	.....	5.08	98.50
Talc No. 7	62.17	0.51	1.43	0.00	32.34	.....	.....	.....	.....	.....	.....	2.98	99.43
Talc No. 8	66.40	28.12	0.71	0.00	0.39	.....	.....	.....	.....	.....	.....	5.10	100.72

[a] Not determined.

[b] Combined oxides of Fe and Al.

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AVERAGE SIZE AND DISTRIBUTION OF PARTICLES.

The average particle sizes as calculated from the total or gross sample, on the basis of surface, are given in Table 4 (p. 15), as are also the averages calculated from the samples elutriated. As previously stated, these figures do not give the distribution of the different sizes, and the grains might all be of the average size without this fact being disclosed by the statement of the average. To show this distribution Tables 10 and 11 have been calculated. These give the percentage of the different sizes by surface and by weight. For convenience the 2-micron and smaller grains have been grouped as of 1-micron size. Distribution based on the frequency or number of particles would have little meaning with nonmetallic fillers, as the range in size is so great and the preponderance of finer sizes so enormous. The calculations for surface percentage are made by dividing the product of the number of each size and its diameter squared by the sum of such product, or $\frac{nd^2}{\Sigma nd^2}$=per cent of surface. Similarly the weight percentages are obtained by dividing the product of the number of each size and its diameter cubed by the sum of these products, or $\frac{nd^3}{\Sigma nd^3}$=per cent of weight.

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TABLE 10.—*Percentage of different sizes based on weight or volume, calculated from count of total sample.*

Sample.	Diameter, microns.												
	Average diameter.	80	60	50	40	30	20	15	10	7	5	3	1
Barite No. 1	9.8	0.0	0.9	5.7	14.0	17.8	13.8	14.7	8.3	9.2	7.1	6.7	1.7
Barite No. 5	7.3	0.0	0.0	0.5	2.1	6.8	16.2	18.3	18.2	14.5	14.8	5.6	2.8
Clay No. 4	2.9	0.0	0.0	3.9	12.1	17.0	10.1	4.2	5.0	7.9	7.4	15.4	16.7
Clay No. 9	3.7	0.0	3.6	1.5	2.6	3.6	13.1	17.7	3.3	8.2	7.5	25.8	13.0
Clay No. 10	5.4	5.1	3.7	4.3	3.0	4.6	12.7	12.7	10.5	9.6	9.9	17.2	6.6
Clay No. 11	3.9	0.0	1.8	3.7	5.2	6.0	24.5	9.2	7.2	6.7	6.5	13.3	15.7
Clay No. 12	5.4	4.7	4.9	4.6	5.9	22.8	11.9	14.7	4.4	4.4	3.2	6.4	12.1
Clay No. 13	5.8	0.0	3.1	3.7	4.7	11.1	13.2	15.2	10.2	9.9	9.5	13.2	6.2
Clay No. 15	6.4	9.2	3.4	3.0	3.4	7.0	12.9	14.5	10.9	12.2	10.2	7.2	5.6
Clay No. 25	3.4	4.8	2.0	3.6	5.6	3.7	5.1	23.8	7.8	3.4	4.5	15.8	19.7
Clay No. 28	5.6	7.1	1.5	4.6	3.8	7.4	8.9	20.0	10.7	8.7	7.5	11.5	7.8
Clay No. 32	3.3	6.0	2.5	3.6	4.5	8.6	5.6	15.9	7.1	5.0	5.2	14.5	21.3
Clay No. 33	5.5	0.0	1.1	3.3	4.2	9.2	15.1	17.6	10.9	10.0	9.2	11.7	7.4
Average of clays		3.3	2.5	3.6	5.0	9.2	12.1	15.0	8.0	7.8	7.3	13.8	12.0
Graphite No. 5	23.4	2.6	32.6	10.5	10.6	9.8	12.5	9.6	6.1	2.9	1.9	0.6	0.3
Whiting No. 1	10.7	0.0	0.0	2.3	4.9	16.0	24.3	19.2	11.8	10.4	6.1	3.9	0.9
Whiting No. 2	5.9	4.8	3.6	2.1	3.2	4.4	11.7	18.4	8.8	9.4	13.2	13.7	6.5
Whiting No. 4	5.7	0.0	0.0	1.6	1.7	3.2	15.7	16.4	13.7	15.9	12.0	14.2	5.5
Whiting No. 6	10.8	1.8	2.3	6.8	7.2	13.4	17.5	16.2	13.8	9.0	7.0	3.4	1.5
Whiting No. 7	5.0	1.4	0.0	2.6	2.7	8.0	5.4	11.7	9.9	12.8	21.4	17.3	6.5
Average of whitings		1.6	1.2	3.1	3.9	9.0	14.9	16.4	11.6	11.5	11.9	10.5	4.2
Mica No. 4	13.8	0.0	0.2	10.0	13.4	9.9	11.5	34.9	11.4	4.4	2.2	1.5	0.4
Mica No. 6	11.2	0.0	0.2	0.7	9.9	20.6	13.0	26.4	10.3	8.1	7.0	2.9	0.9
Mica No. 9	8.7	0.0	0.0	0.0	0.0	0.3	29.5	17.6	32.4	8.3	5.1	5.2	1.7
Average of micas		0.0	0.1	3.6	7.8	10.3	18.0	27.8	18.0	6.9	4.8	3.2	1.0
Ocher No. 1	5.0	4.6	2.1	2.5	4.4	8.3	10.2	16.9	12.1	10.7	9.9	10.0	8.6
Ocher No. 3	4.1	7.0	0.0	0.0	2.8	7.1	7.6	11.0	13.5	19.6	13.1	9.7	8.5
Ocher No. 6	6.6	3.3	2.9	5.9	10.2	11.8	10.9	13.7	12.0	9.1	8.8	4.5	6.5
Average of ochers		5.0	1.7	2.8	5.8	9.1	9.6	13.9	12.5	13.1	10.6	8.1	7.9
Slate flour No. 2	9.8	20.8	5.5	5.9	6.4	8.8	8.6	7.2	9.4	11.8	8.4	5.4	1.7
Slate flour No. 3	4.6	0.0	0.0	1.4	1.5	6.7	4.6	4.5	10.4	24.9	23.4	15.9	6.5

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Silica No. 1	8.1	1.1	1.0	1.3	2.9	5.6	14.2	23.9	16.4	14.6	9.3	7.5	1.9
Silica No. 6	5.2	16.0	0.0	4.1	4.2	5.3	7.4	6.2	13.9	18.2	9.4	7.9	7.2
Silica No. 8	5.9	8.1	2.3	4.0	3.5	6.3	9.0	9.9	14.6	15.1	11.6	10.0	5.4
Average of silicas		8.4	1.1	3.1	3.5	5.7	10.2	13.3	15.0	16.0	10.1	8.5	4.8
Talc No. 2	12.4	0.3	3.9	16.5	8.0	8.6	11.9	15.9	21.9	4.6	4.4	2.7	1.2
Talc No. 5	12.6	6.7	6.3	6.7	3.8	14.6	9.4	14.8	15.5	11.0	5.6	4.6	0.9
Talc No. 7	8.7	1.8	1.3	3.1	5.9	13.5	15.3	14.1	15.7	9.6	9.6	6.6	3.5
Talc No. 8	11.8	0.3	2.5	7.9	7.2	9.1	31.4	15.4	10.2	8.4	3.5	3.3	0.8
Average of talcs		2.3	3.5	8.5	6.2	11.4	17.0	15.1	15.8	8.4	5.8	4.3	1.6

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TABLE 11.—*Percentage of different sizes based on surface, calculated from count of total sample.*

Sample.	Diameter, microns.												
	Average diameter.	80	60	50	40	30	20	15	10	7	5	3	1
Barite No. 1	9.8	0.0	0.1	1.1	3.4	5.8	6.7	9.6	8.1	12.8	13.9	21.7	16.6
Barite No. 5	7.3	0.0	0.0	0.1	0.4	1.7	5.9	8.9	13.2	15.1	21.5	13.7	19.5
Clay No. 4	2.9	0.0	0.0	0.2	0.9	1.7	1.5	0.8	1.5	3.3	4.3	15.1	70.6
Clay No. 9	3.7	0.0	0.2	0.1	0.2	0.4	2.4	4.4	1.2	4.4	5.6	32.2	48.6
Clay No. 10	5.4	0.3	0.3	0.5	0.4	0.8	3.4	4.5	5.6	7.3	10.6	30.7	35.4
Clay No. 11	3.9	0.0	0.1	0.3	0.5	0.8	4.8	2.4	2.8	3.7	5.1	17.5	61.8
Clay No. 12	5.4	0.3	0.4	0.5	0.8	4.1	3.2	5.3	2.3	3.3	3.5	11.5	64.8
Clay No. 13	5.8	0.0	0.3	0.4	0.7	2.1	3.8	5.9	5.9	8.2	11.0	25.5	36.0
Clay No. 15	6.4	0.7	0.4	0.3	0.5	1.5	4.1	6.2	6.9	11.1	13.1	15.3	39.6
Clay No. 25	3.4	0.2	0.1	0.2	0.5	0.4	0.9	5.1	2.7	1.8	3.1	17.9	66.9
Clay No. 28	5.6	0.4	0.1	0.5	0.5	1.4	2.5	7.4	6.0	7.0	8.3	21.6	44.1
Clay No. 32	3.3	0.2	0.1	0.2	0.4	0.9	0.9	3.5	2.3	2.3	3.4	15.8	69.9
Clay No. 33	5.5	0.0	0.1	0.4	0.6	1.7	4.2	6.5	6.0	7.9	10.2	21.6	40.9
Average of clays	.....	0.2	0.2	0.3	0.5	1.4	2.9	4.7	3.9	5.5	7.1	20.4	52.6
Graphite No. 5	23.4	0.8	12.6	5.4	6.5	7.6	12.9	14.8	12.5	8.9	8.6	3.4	6.1
Whiting No. 1	10.7	0.0	0.0	0.5	1.3	5.7	12.9	13.7	12.6	15.9	13.2	14.1	10.0
Whiting No. 2	5.9	0.3	0.4	0.2	0.5	1.0	3.8	7.9	5.8	8.8	14.7	30.5	25.9
Whiting No. 4	5.7	0.0	0.0	0.2	0.2	0.6	4.4	6.2	7.7	13.0	13.6	26.9	27.2
Whiting No. 6	10.8	0.2	0.4	1.5	1.9	4.8	9.5	11.7	14.9	13.9	15.2	12.3	13.6
Whiting No. 7	5.0	0.1	0.0	0.3	0.3	1.3	1.4	3.9	5.0	9.2	21.5	29.1	27.8
Average of whitings	.....	0.1	0.2	0.5	0.8	2.6	6.4	8.5	9.2	12.2	15.6	22.6	20.9
Mica No. 4	13.8	0.0	0.1	3.2	5.0	4.9	8.2	33.1	16.1	9.2	6.3	7.1	6.8
Mica No. 6	11.2	0.0	0.1	0.2	3.1	8.9	6.3	18.9	9.9	10.5	16.0	12.9	13.1
Mica No. 9	8.7	0.0	0.0	0.0	0.0	0.1	13.6	11.2	27.2	10.6	9.7	15.4	12.2
Average of micas	.....	0.0	0.1	1.1	2.7	4.6	9.4	21.6	17.7	10.1	10.7	11.8	10.7
Ocher No. 1	5.0	0.2	0.2	0.2	0.6	1.3	2.5	5.6	6.0	7.7	9.8	16.7	49.1
Ocher No. 3	4.1	0.3	0.0	0.0	0.3	1.0	1.6	3.0	5.5	11.5	10.8	13.2	52.8
Ocher No. 6	6.6	0.3	0.3	0.8	1.7	2.6	3.6	6.1	8.0	8.6	11.6	10.0	46.3
Average of ochers	.....	0.3	0.2	0.3	0.9	1.6	2.6	4.9	6.5	9.3	10.7	13.3	49.4
Slate flour No. 2	9.8	1.7	1.0	1.3	1.7	3.1	4.6	5.1	10.1	18.1	18.1	19.3	15.9
Slate flour No. 3	4.6	0.0	0.0	0.1	0.2	1.0	1.1	1.4	4.8	16.6	21.7	24.5	28.5

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Silica No. 1	8.1	0.1	0.1	0.2	0.6	1.5	5.7	13.0	13.4	16.9	15.1	20.1	13.1
Silica No. 6	5.2	0.7	0.0	0.4	0.5	1.0	1.9	2.2	7.3	13.5	9.8	14.2	48.5
Silica No. 8	5.9	0.5	0.2	0.5	0.5	1.2	2.6	3.9	8.5	12.7	13.6	19.6	36.1
Average of silicas		0.4	0.1	0.4	0.5	1.2	3.4	6.4	9.7	14.4	12.8	18.0	32.6
Talc No. 2	12.4	0.1	1.0	4.1	2.7	3.5	6.1	10.7	26.0	9.6	13.1	11.3	11.6
Talc No. 5	12.6	0.7	1.1	1.7	1.1	5.9	5.2	10.9	17.2	16.3	12.4	16.9	10.4
Talc No. 7	8.7	0.2	0.2	0.5	1.2	3.7	6.3	8.7	12.9	11.8	16.8	16.4	21.2
Talc No. 8	11.8	0.0	0.5	2.3	2.3	3.9	18.2	13.2	13.4	14.2	8.9	14.1	9.0
Average of talcs		0.2	0.7	2.1	1.8	4.2	9.0	10.9	17.4	13.0	12.8	14.7	13.0

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To show graphically the difference in distribution between fillers of different minerals the average percentages of surface and weight for the different materials were plotted, the averages of each

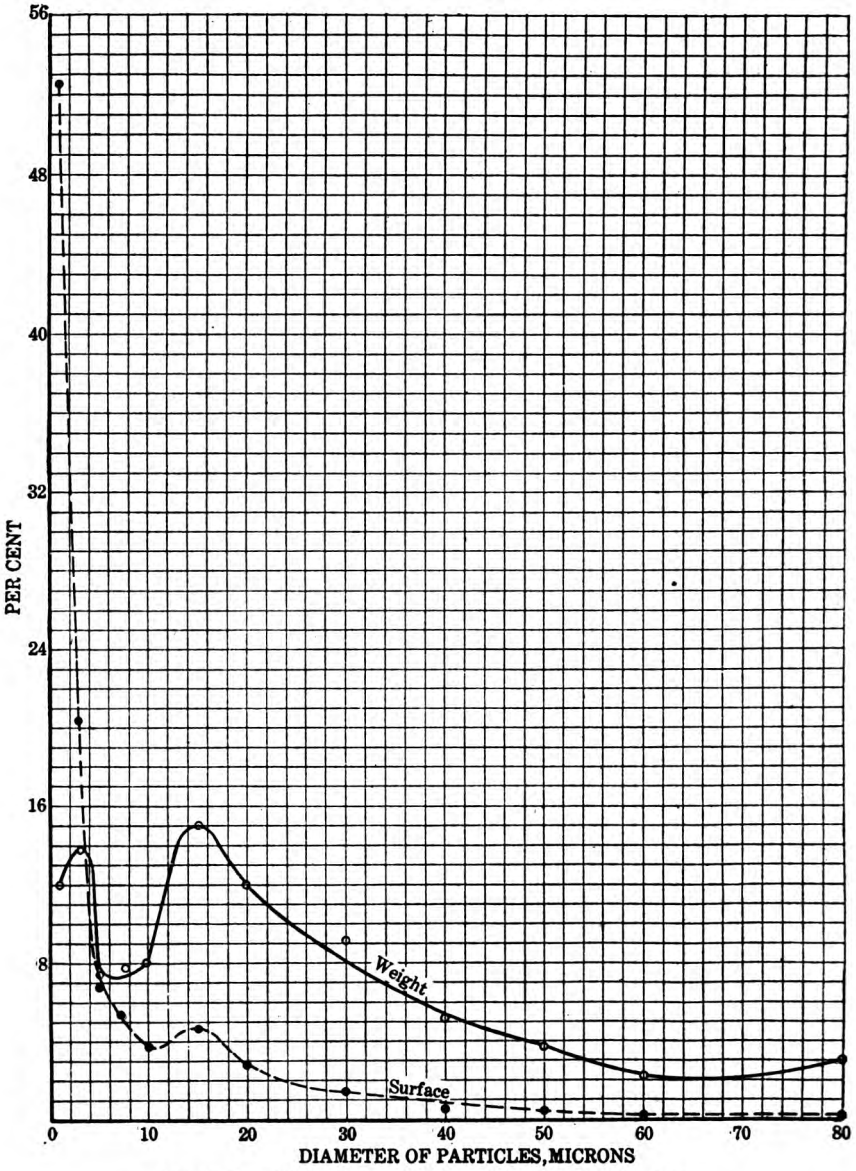

FIGURE 1.—Comparison of different-sized grains in clays.

mineral by each method on the same chart. (See Figs. 1, 2, 3, 4, 5, and 6.)

With the exception of silica, a noteworthy similarity is that the maximum per cent by weight is around 15 microns. Moreover, the weight per cent of all samples drops off in the finer sizes after reach-

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ing a maximum. The drop is less pronounced, however, with clays, and the curve for clay and that for whitings show a peculiar drop, difficult to explain, between the maximum and minimum on the side of the finer sizes. A plausible explanation of this drop, but one not easily proved, is that, due to the structure of some minerals, a reduction in size below 15 microns would tend to make the larger grains break up into particles of 5 microns or less rather than into 8 or 12 micron grains. A point brought out by the surface percentage curves is that when the fineness of grain of minerals is due to natural disintegration or weathering of rocks (clay, ocher, and the silicas being considered) the percentage of surface rises very rapidly in the finer sizes, always showing a maximum for the finest grains considered or those visible with the magnification used, whereas the

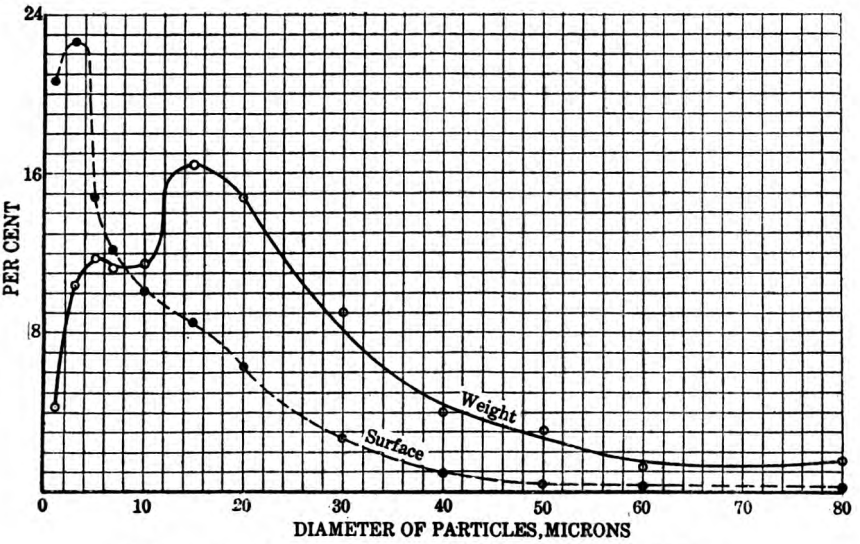

FIGURE 2.—Comparison of different-sized grains in whitings.

fillers produced by mechanical reduction or grinding show a drop in the surface curves in the finer sizes.

PHOTOMICROGRAPHS.

The photomicrographs (Plates II to XIV, p. 44) shown were made to illustrate the physical character of the grains of the different fillers, and the great variation in size of the grains in any one filler, more clearly than pages of figures. They also indicate quite plainly the difficulty of obtaining elutriated fractions uncontaminated by finer grains. It is practically impossible to obtain one field for photographing that will be truly representative of the distribution of the sizes in a total sample. In the plates that show a magnification of 250 diameters, the large squares represent 75 microns to a side and the small squares 15 microns. At 500 diameters the values are 35 and 7 microns, respectively, and at 1,000 diameters 16 and 3.2 microns.

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At 250 diameters the large squares approximately represent in size the nominal opening in a Tyler Standard 200-mesh testing sieve, or 0.074 mm. Because of the colorless, transparent grains of most nonmetallics it is very difficult to obtain photomicrographs with good contrast, and, on account of the range in size, to show sharp definition of all grains at a fairly high magnification.

CHARACTER OF GRAINS.

When pulverized, each mineral, of course, yields a product whose grains have definite physical characteristics. There is, however, often a decided difference between the grains of fillers from crude

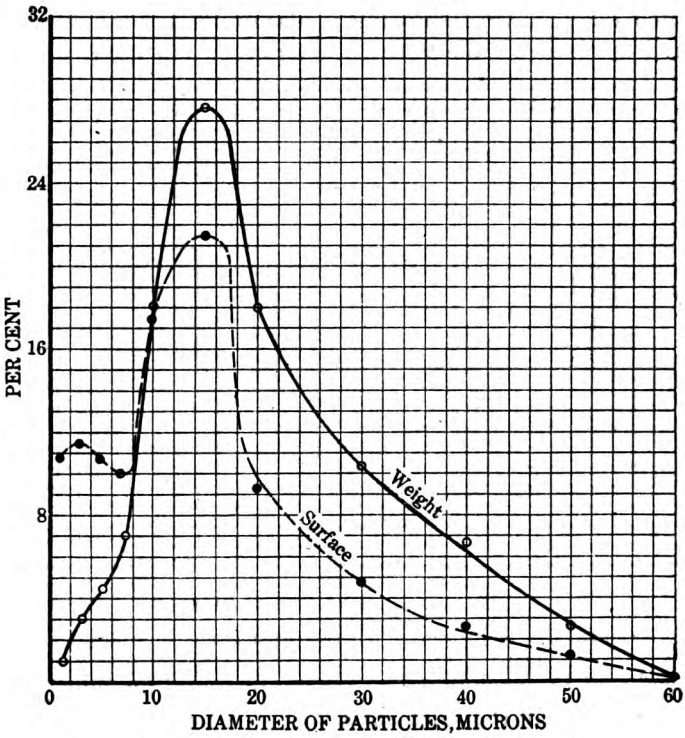

FIGURE 3.—Comparison of different-sized grains in micas.

materials that are similar and have approximately the same composition.

The shape of the grains depends on the crystalline structure of the mineral. This structure also controls the mineral's cleavage and fracture, and its hardness and toughness or resistance to reduction. Of these, the crystalline structure or cleavage is most important. The nonmetallic fillers may be divided into two classes, with respect to the method of reduction or production of the finely powdered product. The first class includes those products that, as mentioned before, are the result of weathering, disintegration, or alteration of other rocks or minerals; this includes clays or kaolins, ocher, and

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umber, and the silicas derived from the weathering of chert or siliceous limestone. Although some of those materials as found in the deposit are rather coherent and strong, they usually break down to their original grains by simple agitation in water. Sometimes

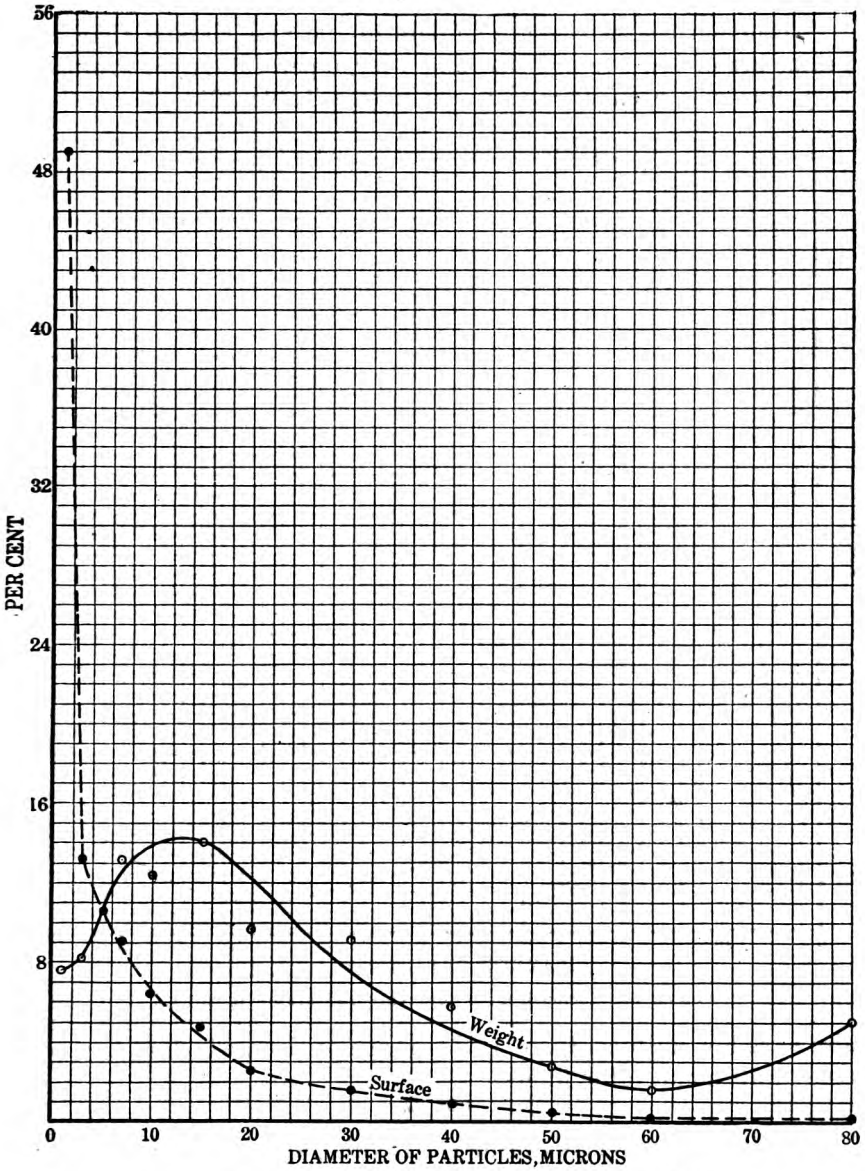

FIGURE 4.—Comparison of different-sized grains in ochers.

they are prepared by grinding, but the grinding is to break up the lumps and aggregates rather than to crush the individual particles and crystals. The second class includes those products that are reduced or ground mechanically, and could not be powdered without con-

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siderable force being applied as pressure or impact. This class comprises barite, whiting, mica, talc, graphite, slate, silica from quartz, and similar materials.

FILLERS WITH NATURALLY FORMED PARTICLES.

A common characteristic of fillers of the first class is that their grains are of low average size, and a large percentage of grains are very fine and even ultramicroscopic (see Table 10). These fillers

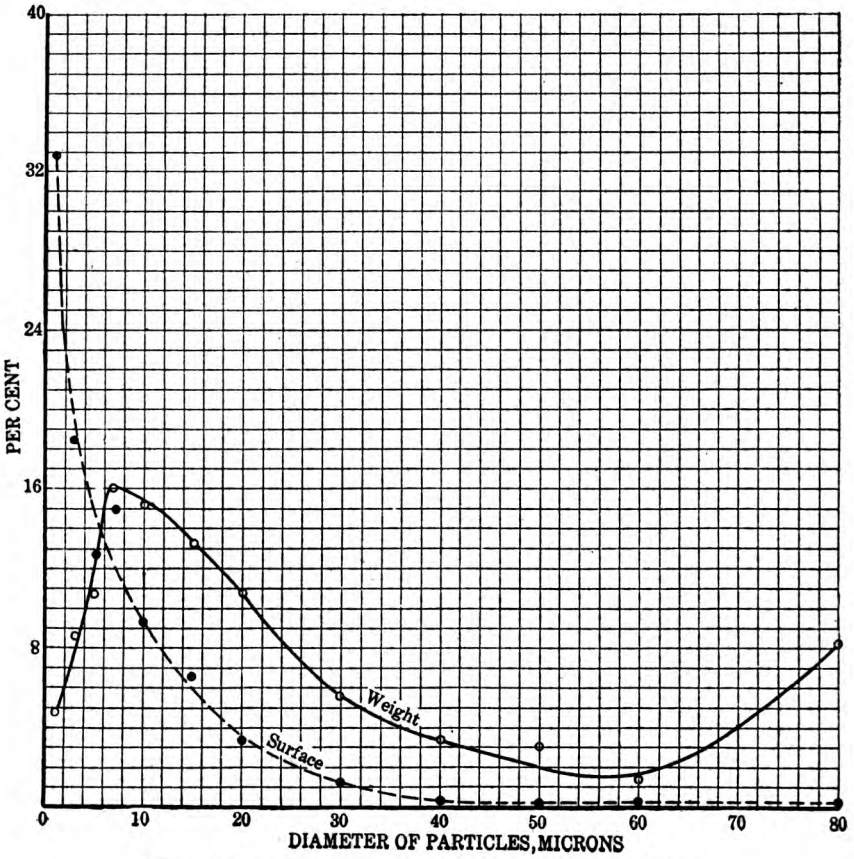

FIGURE 5.—Comparison of different-sized grains in silicas.

also have low uniformity, or a wide range of sizes, which is, however, true of all nonmetallic fillers.

CLAYS.

The sedimentary clays considered in this paper, with the exception of No. 28, have both rounded and angular grains, with the rounded grains predominating. The larger sizes, which are the more angular, include quartz, feldspar, and mica, in addition to kaolin, as shown clearly by Plate V, *a*. The large fragment in the center is mica; the transparent grain in the upper right-hand corner is quartz; and the two dark grains in upper left-hand corner are kaolinite.

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The other photomicrographs of clays show that, as far as the finer particles are concerned, the individual grains are usually crystals or parts of crystals and not aggregates. The larger grains are more often aggregates that are easily broken down by pressure. The intermediate size, rounded grains are most clearly shown in Plates III, *b*, and IV, *a*. The grains are colorless and transparent, except the flocculated particles which are translucent.

OCHER.

The particles of ocher are rounded and are usually aggregates. The impurities, mainly mica and quartz, appear in grains of their customary form. Ocher grains of the finer sizes are translucent.

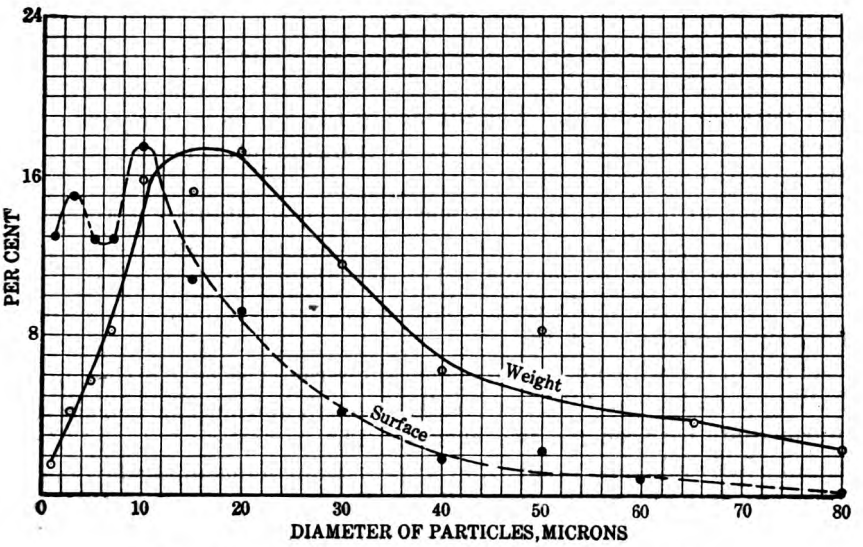

FIGURE 6.—Comparison of different-sized grains in talcs.

Plate VI, *b*, shows the presence of large quartz grains. The fine grains are usually free of impurities, as may be seen in Plate VI, *a*.

SILICA.

All of the samples of silica counted and measured were formed from the weathering of other rocks and may be classed as tripoli.[12] They are all more or less similar in grain structure. The coarser particles are hard, coherent aggregates of finer grains of rounded and botryoidal or incrustated forms. Occasional angular grains of quartz are seen, as just below the central large square in Plate VII, *a*. The finer particles are individual grains, as seen in Plate VII, *b*, and are colorless, transparent, and decidedly rounded.

[12] Ladoo, Raymond B., The mining and preparation of tripoli: Reports of Investigations, Serial No. 2190, Bureau of Mines, Nov., 1920, 9 pp.

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Though the grains are of low average size, as in clay and ocher, ultramicroscopic particles are fewer.

FILLERS WITH ARTIFICIALLY FORMED PARTICLES.

The common characteristics of the second class of fillers are that their grains have a higher average size than those of fillers of the first class and comprise far fewer grains of ultramicroscopic proportions. It is possible so to reduce them by intensive grinding that the average is brought down to 5 microns or less, but even then the proportion of sizes between 2 and 5 microns will tend to increase in a greater ratio than that of sizes below 2 microns. In general, the grains are more angular and rough than those of the naturally formed pigments.

WHITING.

Marble flour is typical of this group. In Plate VIII, *a*, intermediate-sized grains predominate, and the angular form of even the finer particles is evident. The surface of the grains is fairly smooth. They are colorless and translucent to transparent. The crystalline structure is plainly visible.

Whiting ground from limestone shows less angular grains, although the crystalline structure is still evident. Its grains are less smooth than those of marble. The sample counted was from an oölitic limestone, but since the coarsest grains were much finer than the oölites no trace of the characteristic form of the oölites was observed in the whiting. In its general characteristics, whiting from chalk approaches the fillers naturally formed, and may be considered intermediate between the two classes. The particles of the whiting are colorless and translucent; they appear amorphous under low magnification, but are really crystalline. The grains are rounded and their surfaces rough and incrusted, being themselves strongly coherent aggregates of extremely fine individual particles. Plate VIII, *b*, shows the characteristic shape and structure. It is very difficult to disperse the grains for examination on the slide.

BARITE.

Barite much resembles in general appearance the whiting ground from marble. The surfaces of the grains are probably more smooth and they are less angular and sharp than those from marble.

MICA.

Mica flour, or ground mica, has characteristic flat tabular grains, due to the perfect cleavage. The thickness ranges from 10 to 30 per cent of the width. Material ground from schists, chlorite, or sericite has thicker grains than that from muscovite or phlogopite. Plate IX, *a*, a photomicrograph of a chlorite mica, shows the more rounded form, presenting a less perfect cleavage, a ragged fracture, and a less smooth surface than the muscovite in Plate IX, *b*. Here

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the fracture is more in straight lines and the angularity of the grains is very pronounced. The dark spots on the larger grains are due to small grains that lie over or under the large grains and are not inclusions. Plate IX, *b*, shows as wet-ground mica. The dry-ground product is similar, but the edges of the larger flakes are more ragged and the thickness slightly greater. The predominance of the larger grains is evident, although the photomicrographs can not be considered representative.

SLATE FLOUR.

The elutriated fractions of a slate flour are shown in Plates X, *b*, to XII, *b*, inclusive. Rounded grains with rough surface and a tendency to elongated form are in evidence, especially in the larger sizes. The color is slightly green in this sample, and the finer grains are translucent to transparent. Pyrite is visible in grains of less than 1 micron diameter. The larger particles are mostly aggregates of the finer grains seen individually in Plate XII, *b*, their longer dimensions lying parallel with the longer axis of the larger particle. A comparison of Plates XII, *b*, and VII, *b*, shows clearly the difference in proportion of very fine sizes in materials artificially reduced and those naturally reduced from natural products. Both of these were overflow products from the last elutriation can, and were reproduced at the same magnification. Moreover, the specific gravity of the slate is greater than that of the silica. This shows conclusively the possible error in taking an arbitrary value for the average diameter of the different elutriated fractions.

TALC.

Talcs crush to tabular grains. The edges of the larger grains are rough and uneven, but those of the finer grains are smoother. The large particles, made up of aggregates of finer flakes, have a laminated structure and show a rather rough, uneven surface. They are colorless and translucent to transparent. The finer grains are angular, with sharp corners, as shown in Plate XIV. The comparative coarseness of the larger grains is illustrated in Plate XIII. The sample from Winterboro, Ala. (talc No. 7), differs from the others in being more granular and less tabular in structure. The pyrophyllites generally have more rounded grains than the true talcs, but otherwise the grain characteristics are much the same.

GRAPHITE DUST.

The sample of graphite dust here considered is from material that is used more as a dusting powder, or for foundry facing, than strictly as a filler, except possibly in paints. The graphite flakes are quite smooth and have regular edges; they are opaque even in the finest sizes, and are dull black to brown in color. Unlike most of the other products, the impurities, mainly quartz, feldspar, and pyrite, consti-

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tute the majority of the finer grains, 3 microns and less. The flakes are very free from included grains of impurities.

METHOD OF GRINDING.

The method of grinding is not believed to have much effect on the shape or the character of the grains of most fillers. When the majority of minerals are reduced to the fineness of most fillers it becomes impossible to say whether they have been ground by impact, by rolling, or in a tube mill. This does not mean that all methods of grinding are equally efficient in a mechanical way. Wet and dry ground mica are exceptions to this. In wet-ground mica the surface of the large grains is smoother and glossier and the edges are fairly straight lines or curves, but in the dry-ground mica the edges are to a certain extent ragged, as if torn. Dry grinding is generally done by impact or shearing, whereas it is possible that in wet grinding the breaking of the flakes is largely accomplished by bending.

SUMMARY.

In this paper there is given in detail a method for the fairly rapid determination of the average particle size of finely ground minerals, where the limiting range of size is so large, on the one hand, as to make ordinary methods of microscopic measurement impracticable, and, on the other hand, where the particles are nearly all too small for analysis by sieves. The method itself is not entirely new, but has been modified to make it applicable to the materials in question.

A brief outline of various methods of computing average particle size is given, and reasons are advanced for advocating the use of the average based on specific surface as especially applicable to fillers.

The limitations of the elutriation method are pointed out, and the possible use of the method in connection with microscopic count and measurement is shown.

The average diameters, as based on specific surface, for the grains of a number of representative fillers in common use are stated. The list is not by any means complete. It includes only materials now used. Most of the samples are from products now on the market. They are fairly representative of similar materials wherever produced. Tables that show the distribution of the different sizes in any one filler are given. The character of the grains of fillers made from different minerals is also given.

The author believes that a better knowledge of the average size of the particles, the distribution of the sizes throughout the mass, and the character of the surface and shape of the individual grains will result in a more efficient use of the nonmetallic fillers and will often indicate the proper material to be used for a specific purpose without the expense and delay occasioned by having to try a large number in actual compounding or experimental manufacture.

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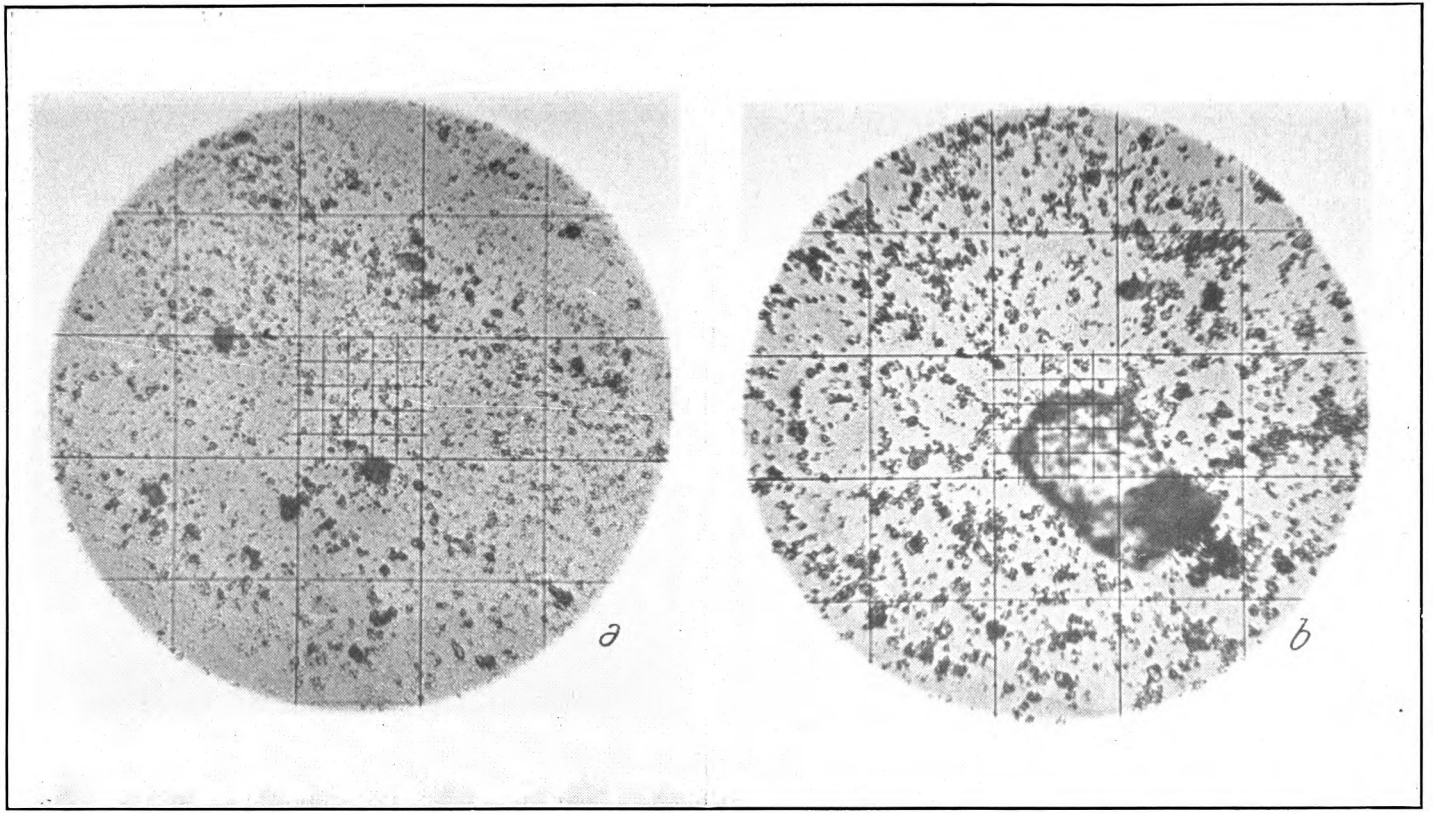

a, CLAY NO. 4, FIFTH ELUTRIATED FRACTION. ×500.

b, CLAY NO. 9. ×500.

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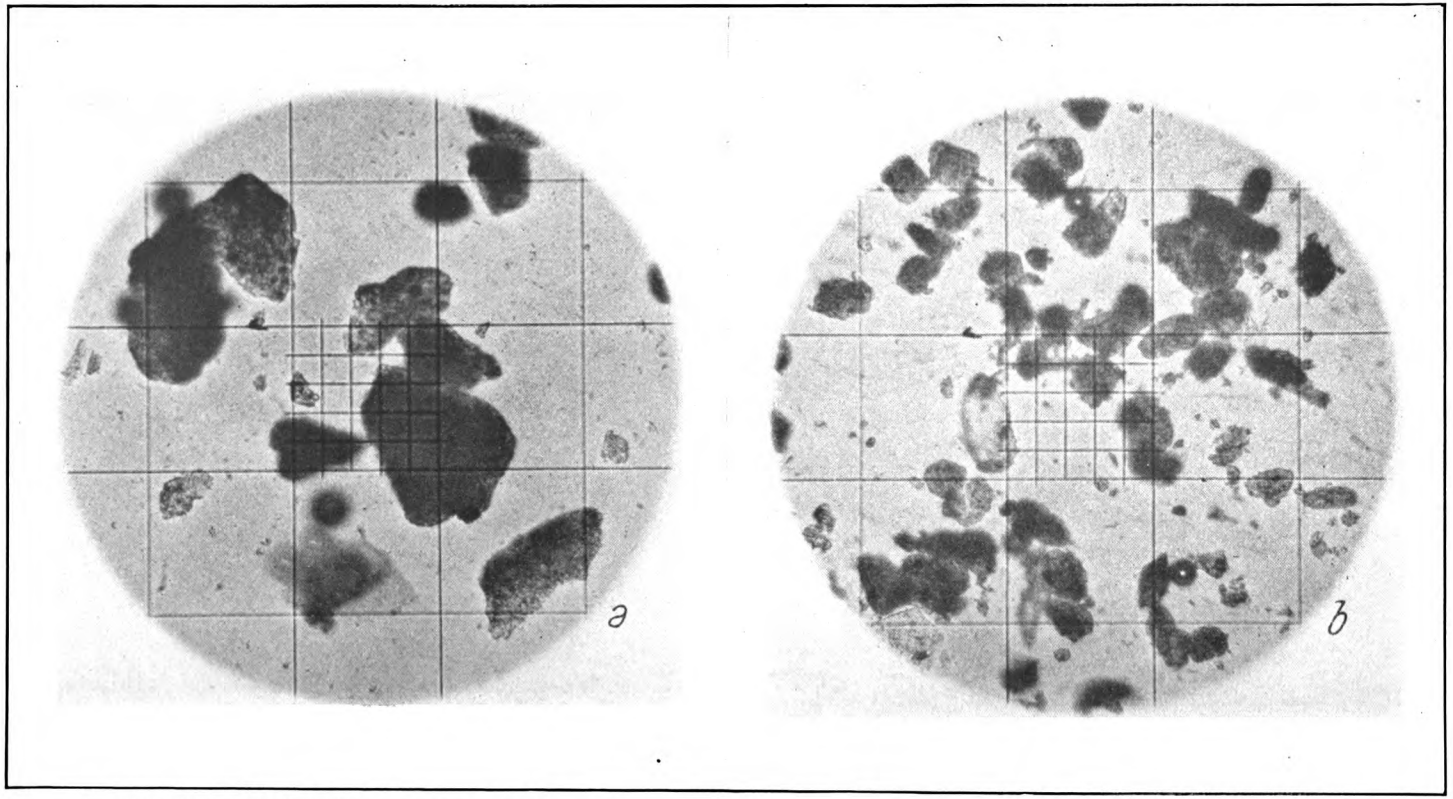

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b, CLAY NO. 9, SECOND ELUTRIATED FRACTION. ×250.

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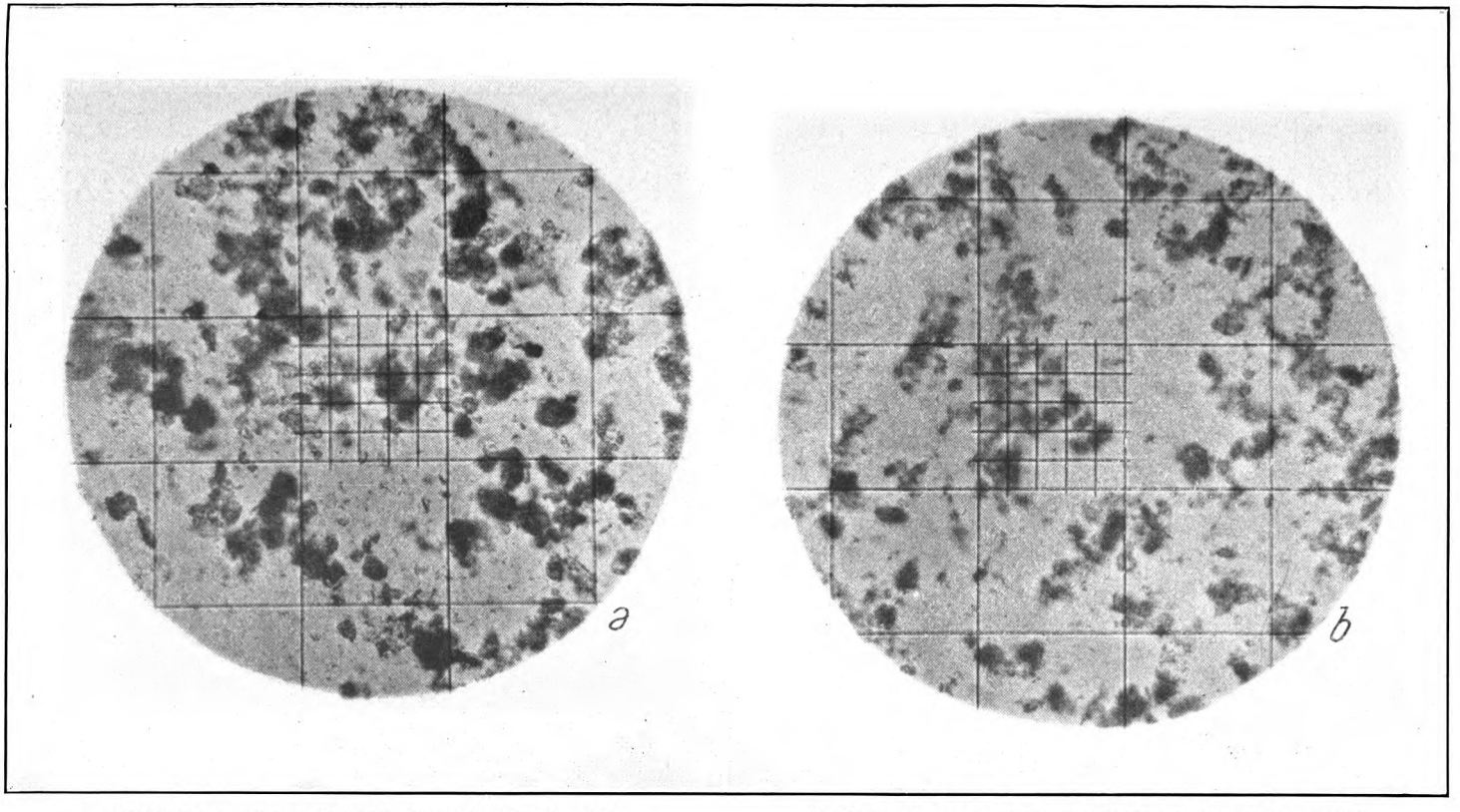

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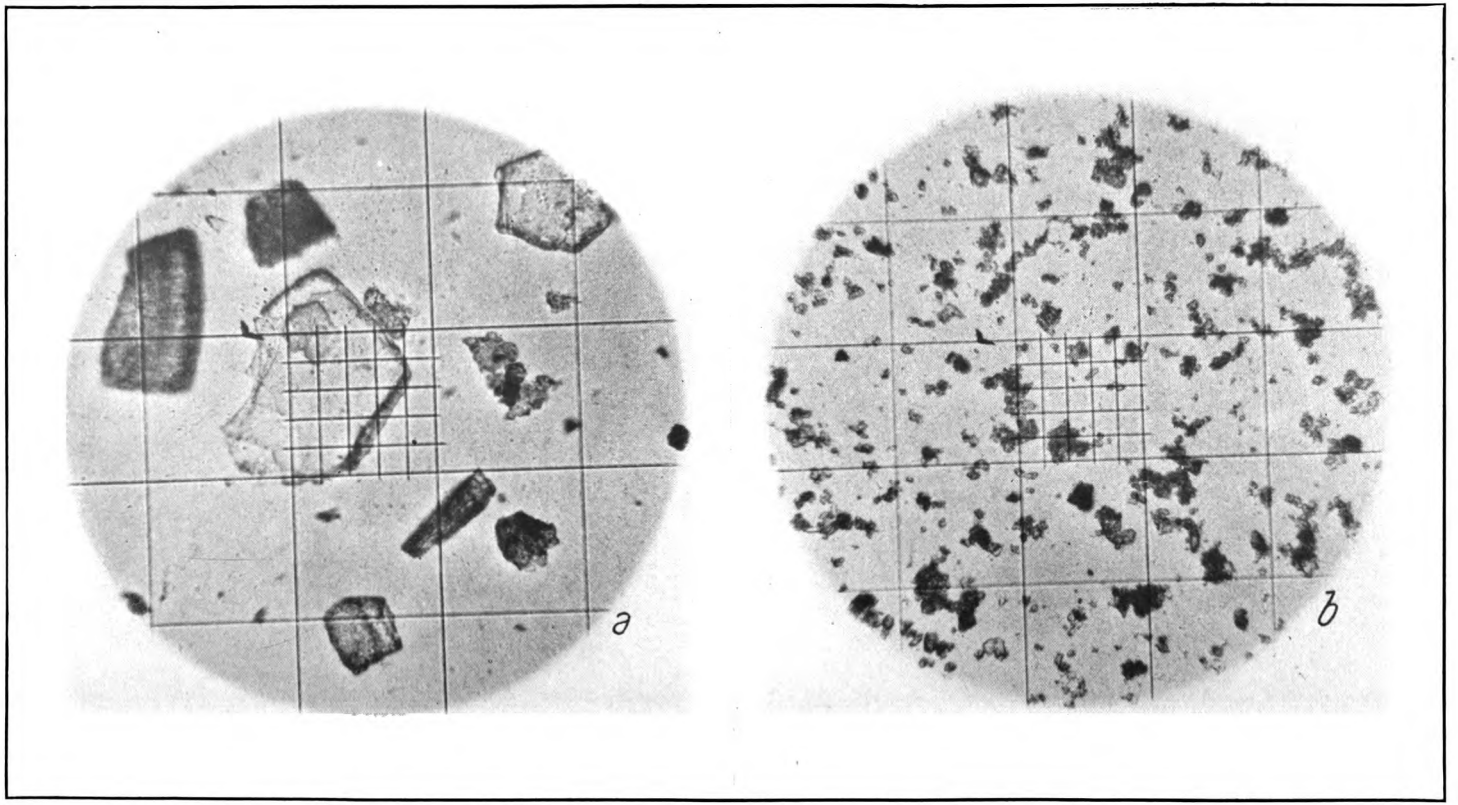

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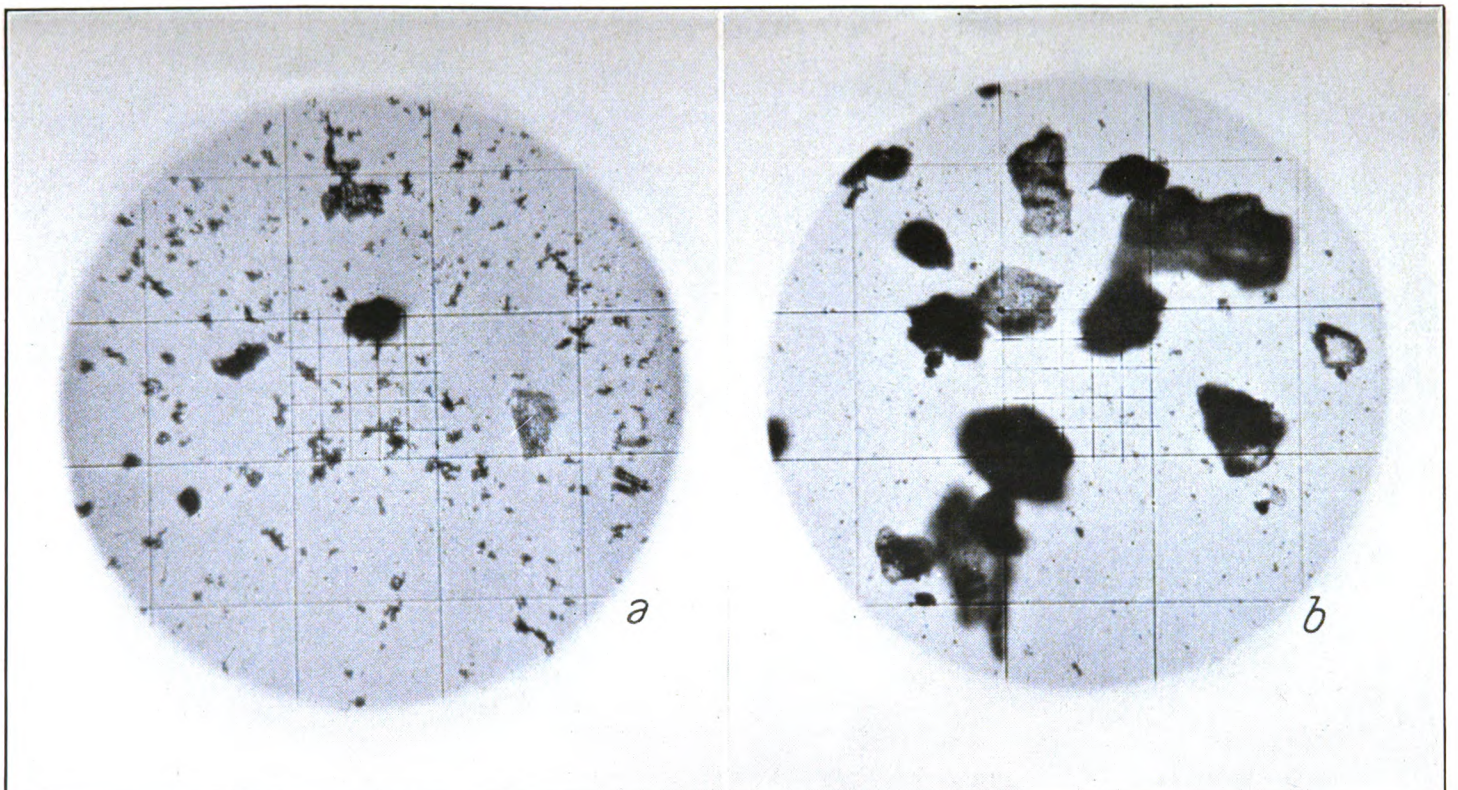

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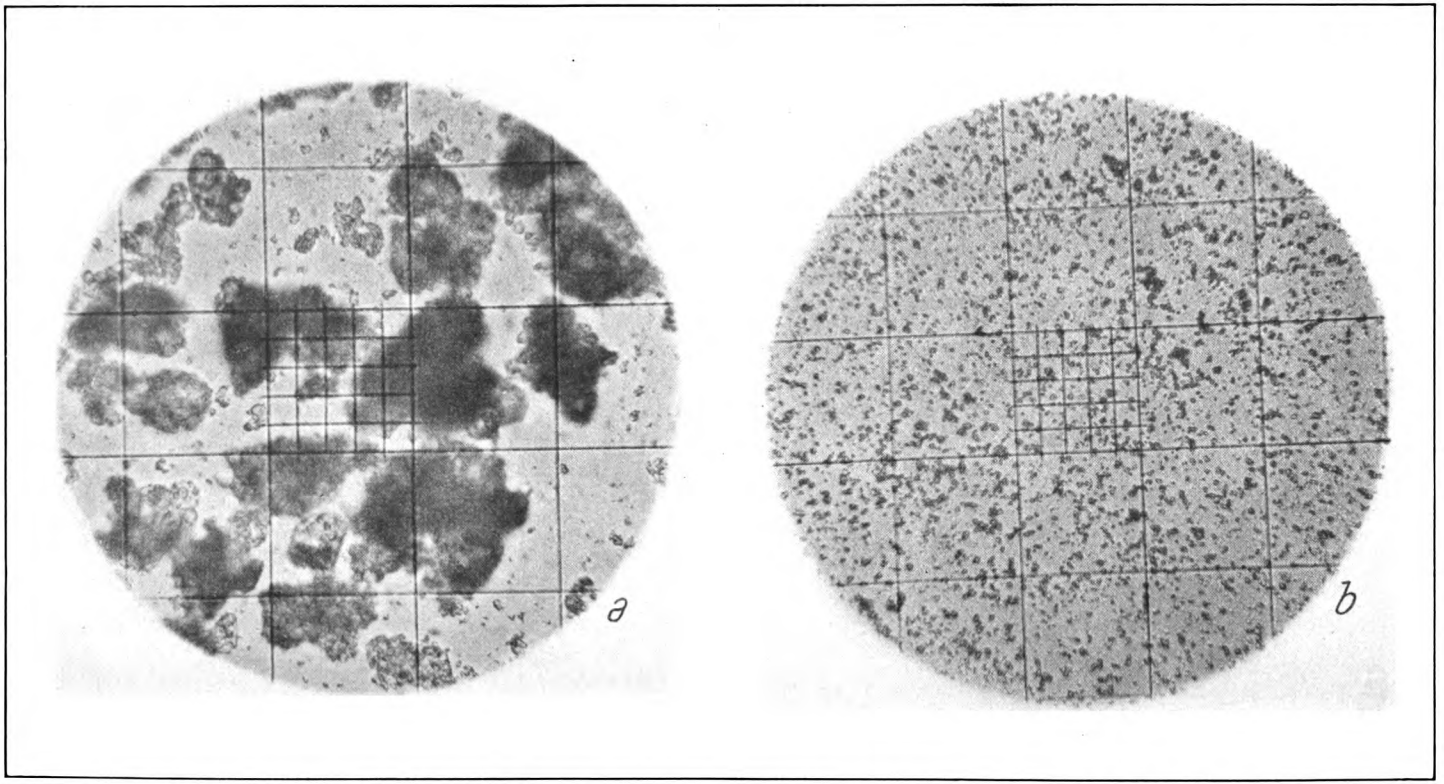

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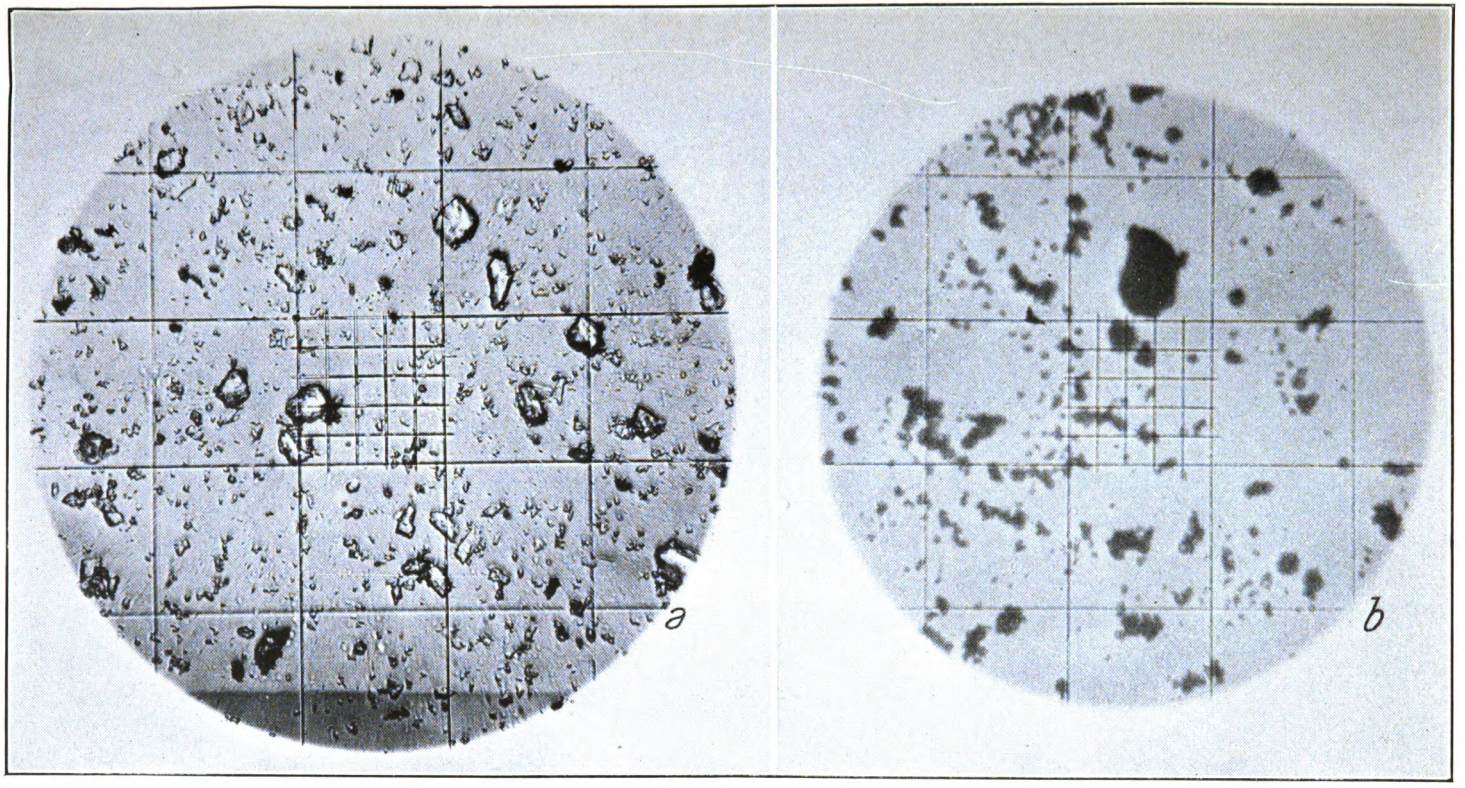

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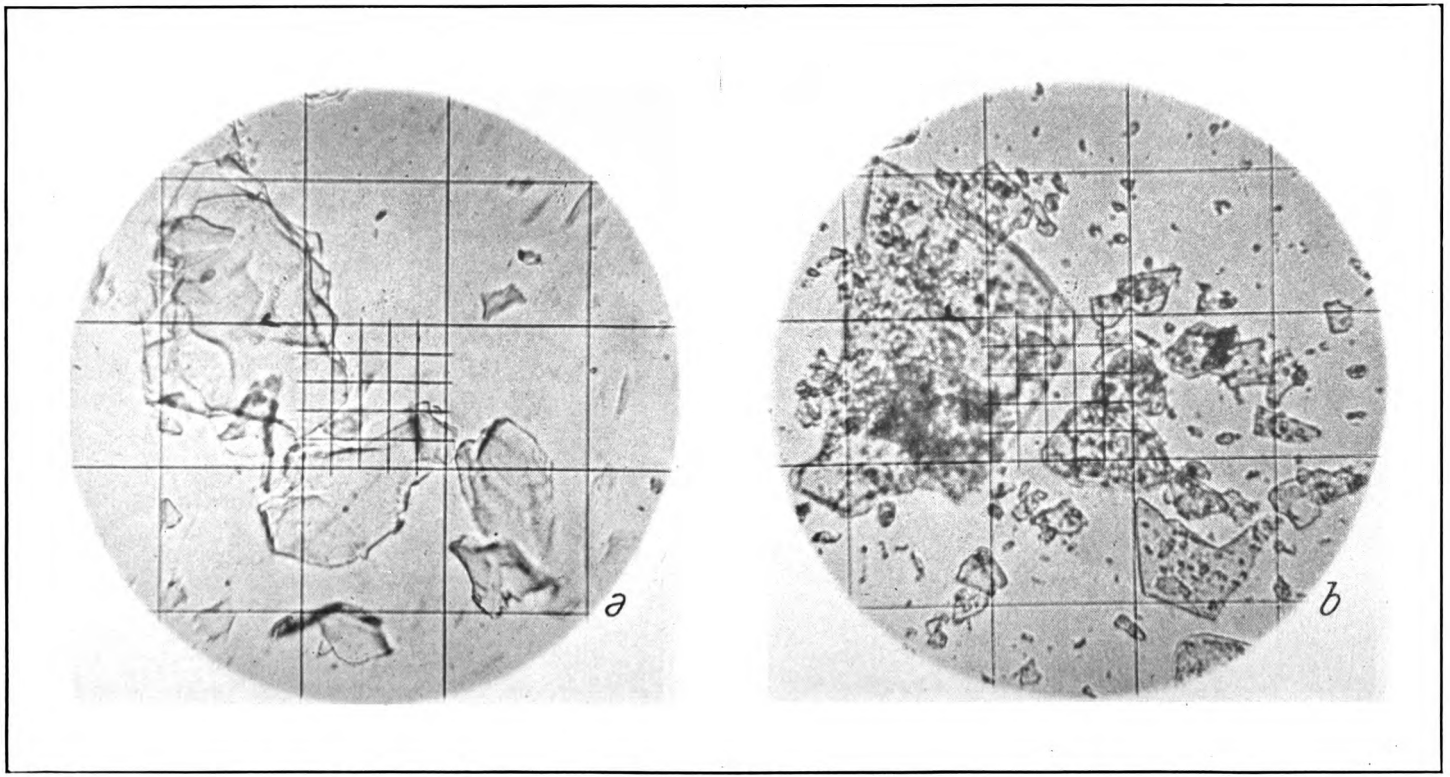

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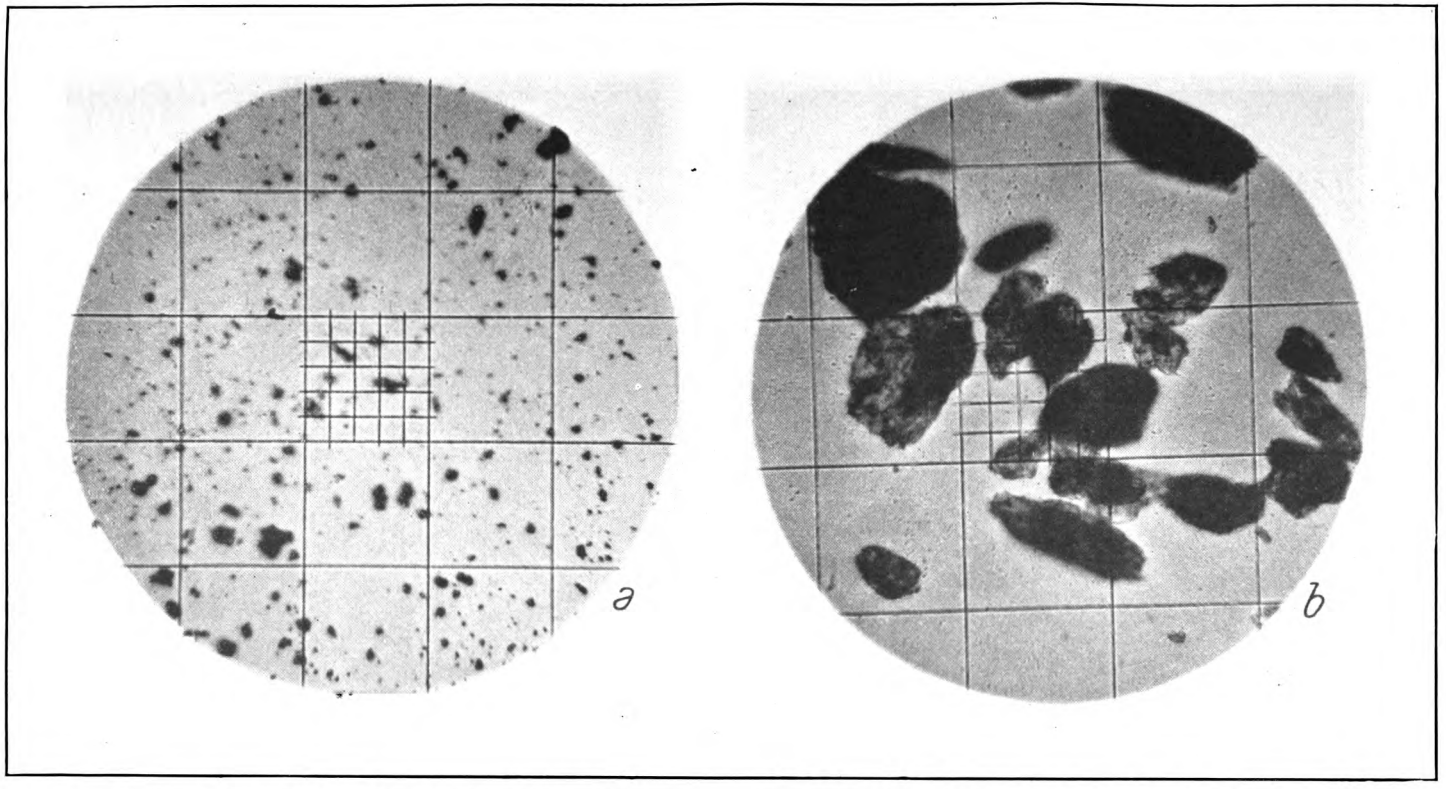

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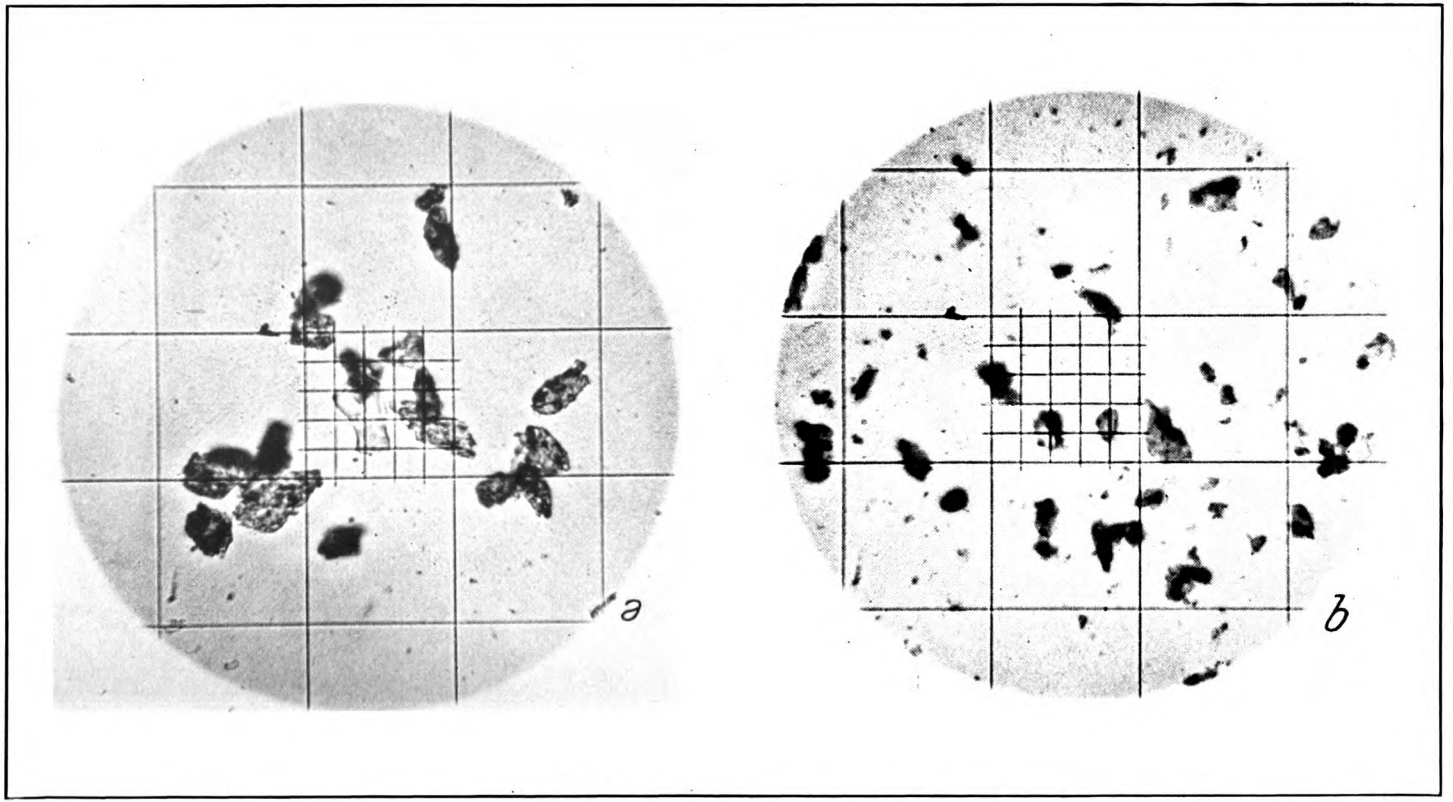

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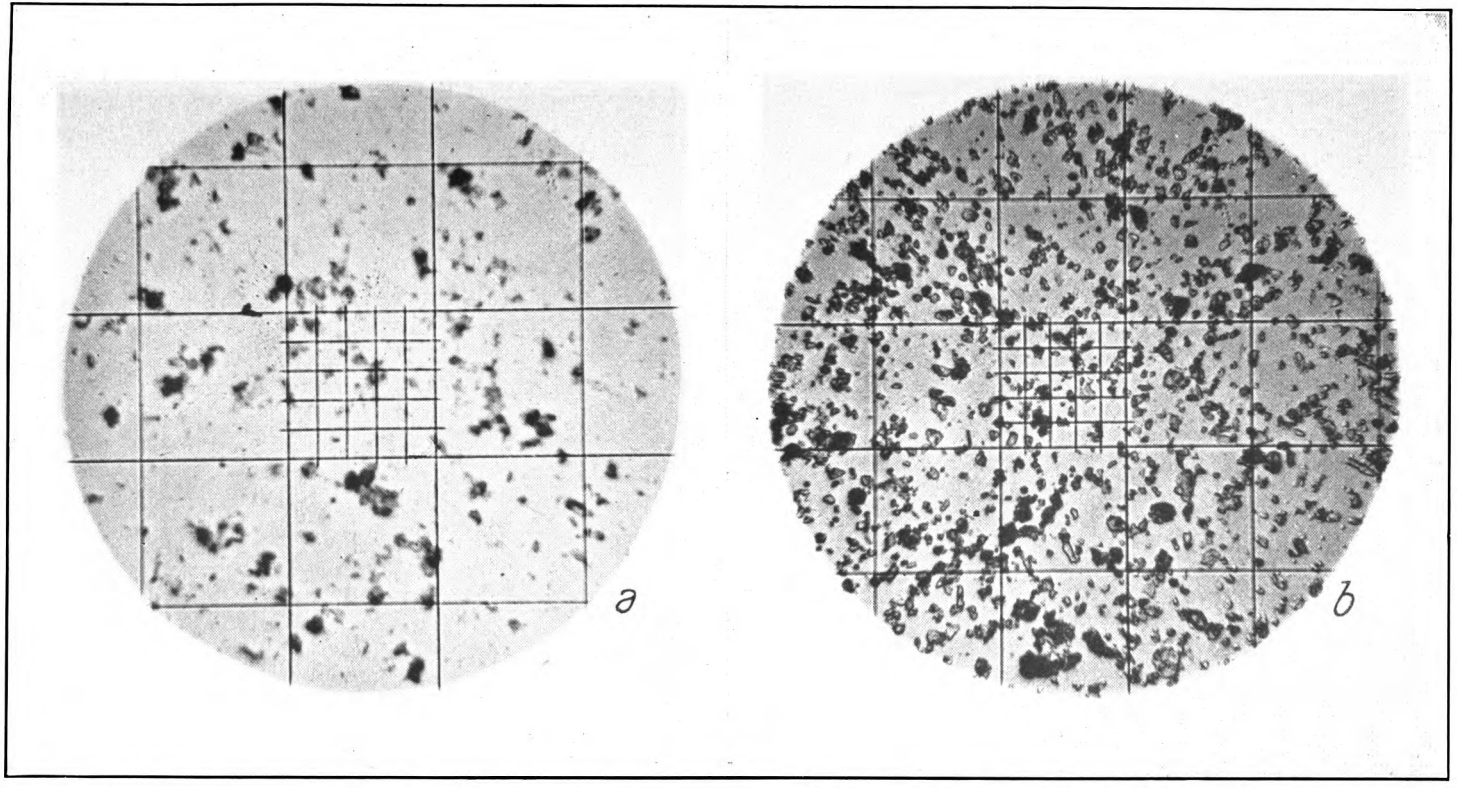

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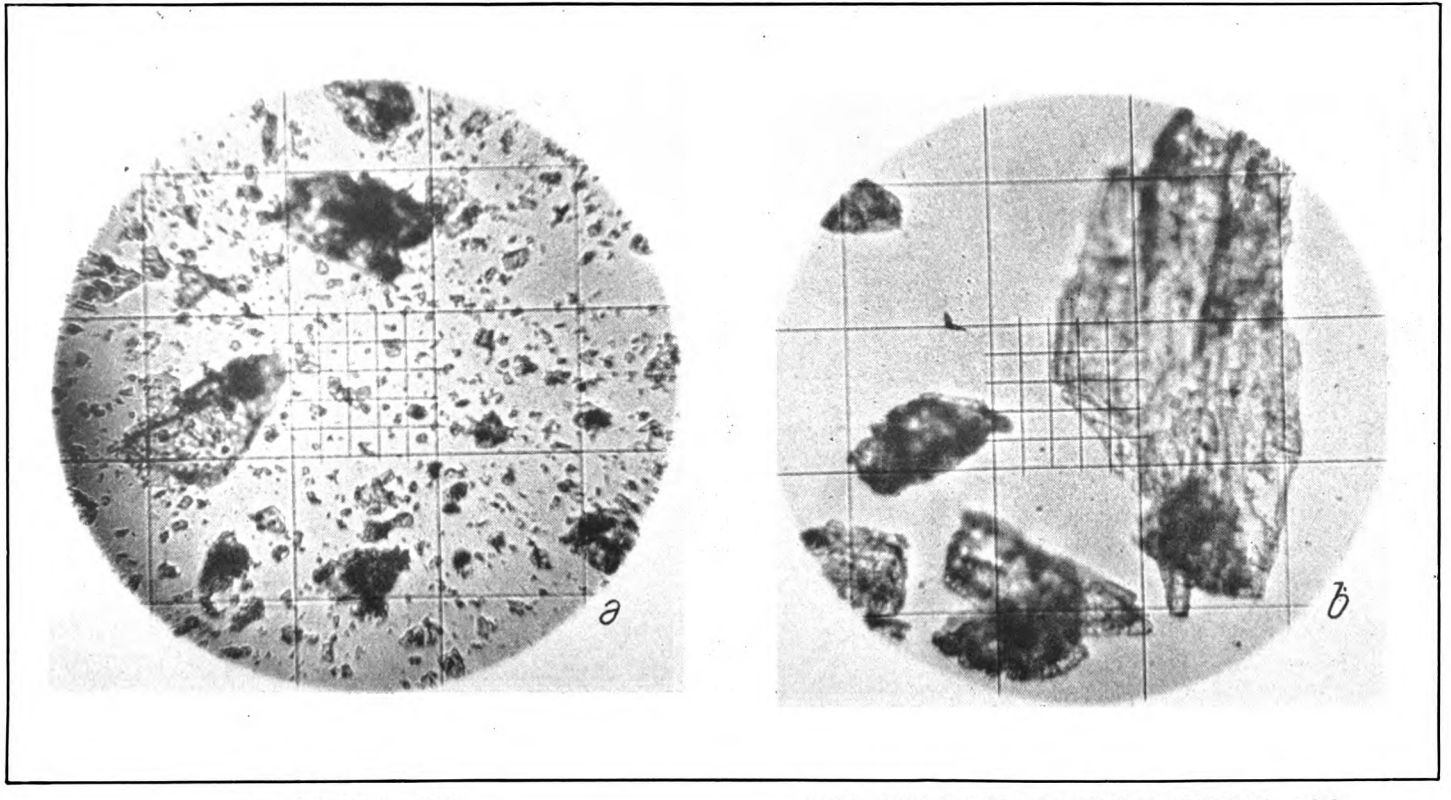

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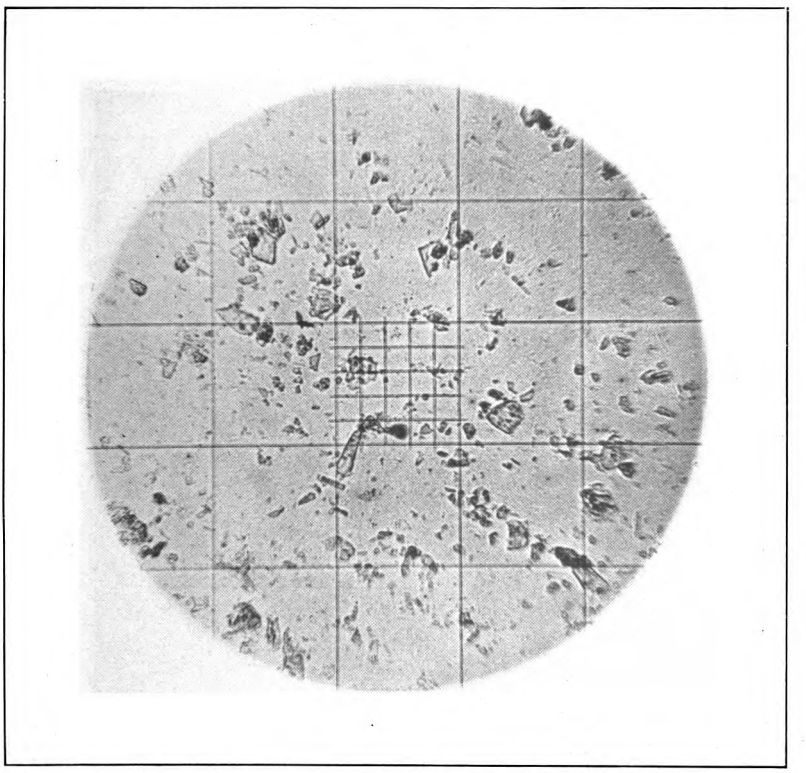

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